DOE/PC/92149--T11

FINAL TECHNICAL REPORT

AUGUST 4, 1992 THROUGH SEPTEMBER 30, 1995

Contract Number DE-AC22-92PC92149

Deashing of Coal Liquids with Ceramic Membrane
Microfiltration and Diafiltration

Submitted by

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To

U.S. Department of Energy

Pittsburgh Energy Technology Center

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EXECUTIVE SUMMARY

Removal of mineral matter from liquid hydrocarbons derived from the direct liquefaction of coal is required for product acceptability. Current methods include critical solvent Deashing and filtration, both of which produce an ash reject stream containing up to 15% of the liquid hydrocarbon product.

This program was directed towards development of an improved process for deashing and recovery of coal-derived residual oil: the use of ceramic membranes for high-temperature microfiltration and diafiltration. Using laboratory scale ceramic membrane modules, samples of a coal-derived residual oil containing ash were processed by crossflow microfiltration, followed by solvent addition and refiltration (diafiltration). Excellent recovery of deashed residual oil was demonstrated. Data from this program were used to develop preliminary estimates for production system capital and operating costs that will be used to assess economic feasibility.

The first objective of this program was to demonstrate technical feasibility of crossflow microfiltration (MF) for removal of mineral matter from a coal derived residual oil. A second objective was to demonstrate technical feasibility of diafiltration of MF concentrate using a hydrocarbon diluent.

The five program tasks included (1) ceramic membrane fabrication, (2) membrane test system assembly, (3) testing of the ceramic membranes, (4) design of a demonstration system using full scale membrane modules, and (5) development of estimates for microfiltration capital and operating costs and assessment of process economic feasibility. Several significant accomplishments were realized during this program including:

- Very effective removal of solids from coal liquids using ceramic membrane filtration.
- Recovery of greater than 90% of coal liquids from atmospheric bottoms by ceramic membrane filtration.
- Demonstration of excellent flux levels suggesting that scaleup for a full size liquefaction plant is feasible.

In addition, membrane flux measurements from both batch concentration and differential trials were used to begin sizing a membrane system of commercial capacity. Initial estimates of both installed plant costs and operating costs were developed to assess process feasibility.

I. INTRODUCTION

Removal of mineral matter from liquid hydrocarbons derived from the direct liquefaction of coal is required for product acceptability. Current methods include critical solvent deashing (Rose® process from Kerr-McGee) and filtration (U.S. Filter leaf filter as used by British Coal). These methods produce ash reject streams containing up to 15% of the liquid hydrocarbon product.

CeraMem proposed the use of low cost, ceramic crossflow membranes for the filtration of coal liquids bottoms to remove mineral matter and subsequent diafiltration (analogous to cake washing in dead-ended filtration) to achieve greater product recovery of coal liquid from the solids concentrate. The use of ceramic crossflow membranes overcomes the limitations of traditional crossflow membranes because of the ability to operate at elevated temperature and to withstand prolonged exposure to hydrocarbon and solvent media. In addition, CeraMem's membrane filters are significantly less expensive than competitive ceramic membranes, due to their unique construction. With these ceramic membrane filters, it may be possible to economically reduce the product losses associated with traditional deashing processes.

A. Membrane Background

General Description of Crossflow Membrane Processes

The process for removal of solids from coal derived liquids in this program is crossflow microfiltration (MF). This is a pressure driven membrane process in which particulates are removed from a feed stream (See Figure 1). In microfiltration, the feed stream is pumped over the membrane surface with a transmembrane pressure differential in the range of 20 to 100 psi. The crossflow velocity is generally 100 to 10,000 times the "perpendicular" velocity or filtration velocity. Retained matter is removed from the system as a fluid solids concentrate. The surface shear at the membrane surface controls the buildup of filter cake (or membrane foulant layer) so that, in principle, a steady state filtration rate is attained. This process is to be contrasted with dead-ended filtration in which no crossflow is present, and a filter cake builds continuously as filtrate is removed.

MF is generally employed for removal of submicron particulate and colloidal matter which would rapidly blind surface filters. Also, MF replaces diatomaceous earth precoat filters because of greater product recovery and the elimination of waste disposal of a voluminous spent precoat cake.

MF can be used in a diafiltration process to increase the recovery of liquid or soluble products from a particulate containing stream. In diafiltration, a solvent is added to the fluid concentrate to dilute the solids so that the fluid can be refiltered to remove additional product in the filtrate along with some of the added solvent. Several cycles of this process can be used to remove essentially all the product in the feed stream and produce a
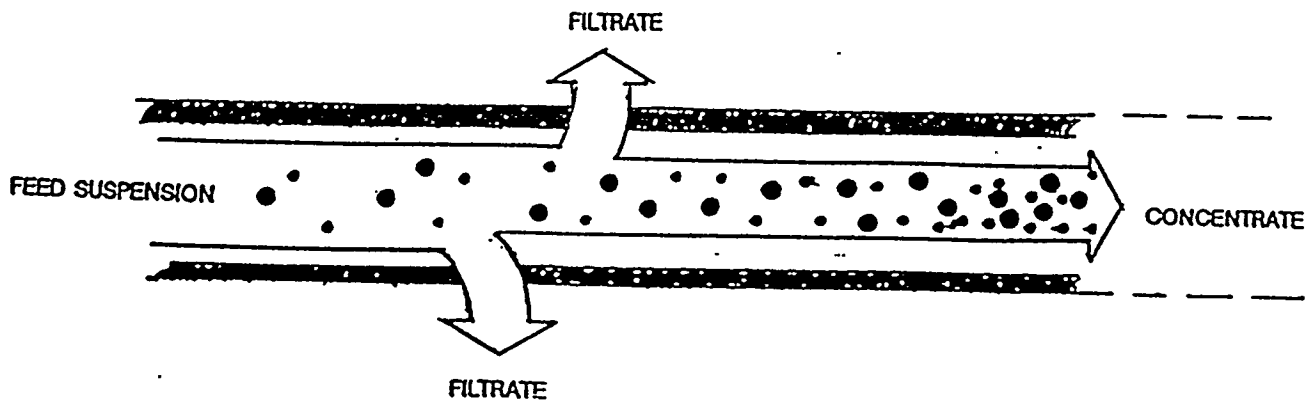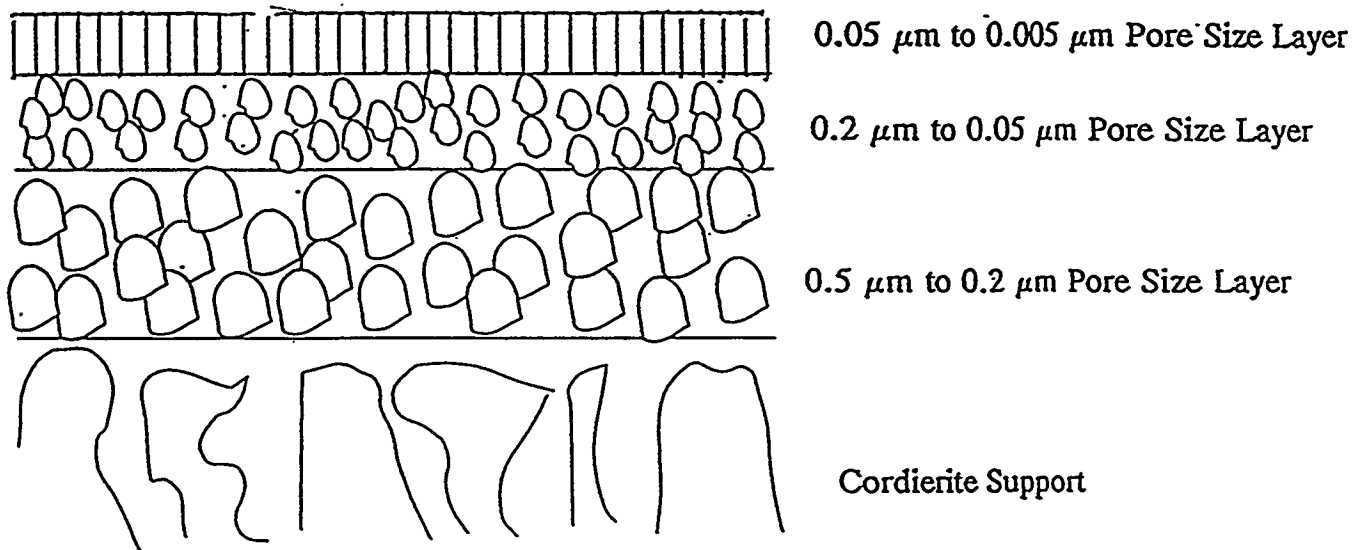
Figure 1.   Crossflow Filtration Schematic

0.05 μm to 0.005 μm Pore Size Layer

0.2 μm to 0.05 μm Pore Size Layer

0.5 μm to 0.2 μm Pore Size Layer

Cordierite Support

Figure 2.   Schematic of Multilayer, Asymmetric Ceramic Membrane

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concentrate consisting of solids and solvent. Depending on the application, additional processing of the permeate and/or concentrate may be necessary to recover the solvent.

Prior Use of Ceramic Membranes for Hydrocarbon Separations

In the late 1970's under DOE contract, the Walden Division of Abcor, Inc. conducted tests to evaluate the applicability of crossflow filtration for the removal of solids from solvent refined coal (SRC) filter feed and solvent deashing overflow [1]. The stainless steel tubular filters used had 1-2 micron pore size. For the SRC filter feed the separation efficiency was excellent, with solids loadings in filtrates below 0.05 wt%. However, initially high crossflow filtration rates declined irreversibly with time. This was attributed to use of filter elements with too large a pore size which allowed the solid particles to enter the pore structure of the filters, irreversibly plugging them. Preliminary tests with solvent deashing overflow (0.25 wt% solids) gave filtrates with very low solids (0.05 wt%) at much higher filtration rates. However, pore plugging was again a problem. It was concluded from this research that a finer pore size crossflow filter would be required for coal liquids processing.

Two recent patents are relevant to the technical approach demonstrated in this program. The first patent [2] discloses two applications in which a ceramic membrane is used [2]. One application involves the use of a 0.02 micron membrane for removal of particulate and other contaminants from waste lubricating oil at a temperature of 200°C. The filtration rate was stable and high because of the elevated operating temperature. The degree of oil purification was very high, with complete removal of colloidal carbon and other fines and high removal of inorganic materials, such as barium, calcium, magnesium, phosphorous, iron, and lead. Based on this work, a 5,000 metric ton per year pilot plant was installed in France about 4 years ago by Total. The major limitation of this work was that high cost (about $200/sq ft) ceramic membranes were employed and the resulting process economics were, therefore, only marginally attractive.

In the process of the second patent [3], a heavy oil, e.g., an asphaltic crude, is hydrotreated in the presence of a finely dispersed (colloidal) catalyst. The product is separated from the catalyst suspension by MF with a ceramic membrane. Different membranes were employed, with pore sizes in the range of 0.015 to 0.3 microns. A 250°C operating temperature was required to reduce feed viscosity. In addition to removal of catalyst for recycle, substantial retention of metals in the concentrate was observed. For example, retentions of nickel and vanadium were about 80% and 90%, respectively.

B. Description of CeraMem's Ceramic Crossflow Membrane Technology

CeraMem is one of several commercial suppliers of liquid crossflow ceramic membrane modules which utilize a porous ceramic monolith as a membrane support. However, CeraMem is unique in its approach in utilizing a very high membrane surface area support structure in each modular element.

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CeraMem's technical approach to construction of ceramic membrane modules is based on the use of porous honeycomb ceramic monoliths as membrane supports. These high surface area, low cost materials have been developed for, and are widely used as, catalyst supports for automotive catalytic converters. The most commonly available material is cordierite. Cell (i.e., feed passageway) "areal densities" in the honeycomb structure range from 9 to 1400 cells per square inch of monolith frontal area (cpsi), and can have round, square, or triangular cell geometries. The porosity of the materials can range from about 30% to 50% with mean pore diameters of 3 μm to 35 μm. The monoliths themselves can be extruded in various cross sections such as rounds, ovals, or squares. Cross sections up to 13" and lengths up to 36" are extruded on a commercial scale by Corning, Inc.

CeraMem forms microfiltration and ultrafiltration membranes on the monoliths by slip casting porous coatings of ceramic particles on the cell wall surfaces of the passageways, followed by drying, and then sintering to bond the particles to each other and the honeycomb support. Most membranes have more than one coating layer, constituting a multilayer, asymmetric ceramic membrane. The initial layers are relatively thick (75-100 μm) and consist of large particles to cover the pores of the support material. Subsequent layers are thinner to minimize flow resistance and consist of finer particles to form finer pore sizes. A schematic diagram of a multilayer membrane is shown in Figure 2 (page 3).

Each monolith has hundreds to thousands of parallel passageways that run from one face to the opposite end face (Figure 3). During processing, the feed stream to be treated is introduced under pressure at one end of the module, flows through the passageways over the membrane, and is withdrawn at the downstream end of the module. Material which passes through the membrane (permeate) flows into the cell walls of the monolith. The combined permeate from all the passageways flows toward the periphery of the monolith support and is removed through an integral, pressure-containing "skin" at the exterior of the monolith.

There is a technical limitation to use of monolith supports as described above. Due to the long and tortuous path through which the permeate must flow to get to the outside skin, there can be a large pressure drop for permeate flow. Depending on membrane resistance and process conditions, the only passageways from which permeate can be effectively removed are often those in an annular ring adjacent to the monolith skin. This limitation generally restricts the diameter of monoliths than can be used to approximately one inch.

CeraMem has developed mechanical modifications to monoliths to overcome this limitation, and one version used commercially for membrane modules is approximately six inches in diameter. These mechanical modifications create permeate conduits within the monolith which conduct permeate from the interior of the monolith to an external permeate collection zone. Figure 4 depicts one form of these mechanical modifications. In this case, slots are cut into one or both ends of the monolith, and the ends of these slots are sealed. At the opposite end of the monolith, the ends of the cells opening into the slots are sealed in a like manner. Many sealants can be used, but the preferred materials are similar to those from which the monolith is made. After sealing the slots at both ends of
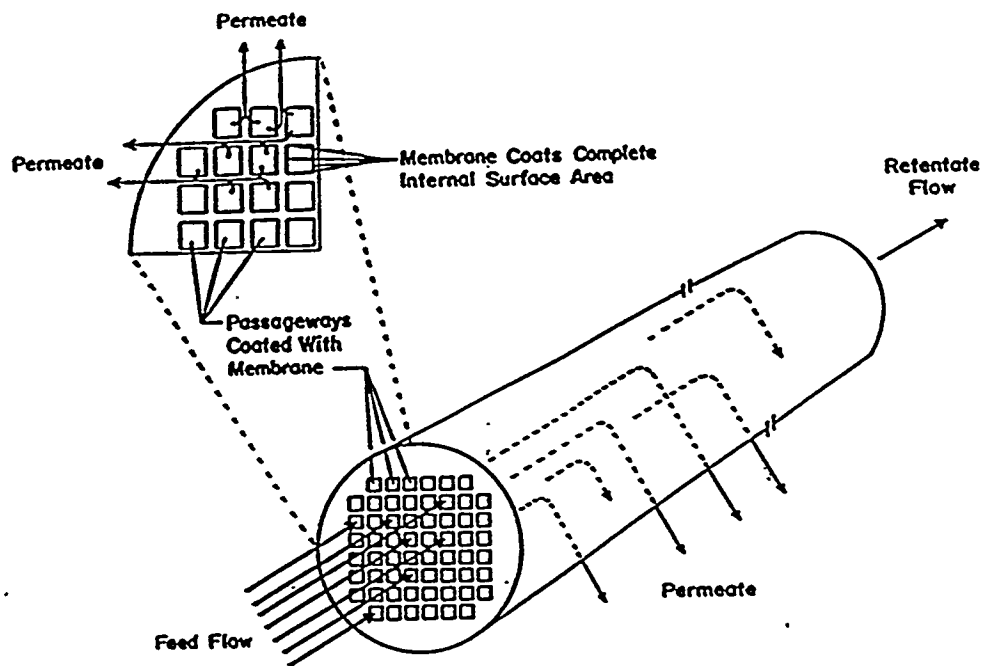
Figure 3.   Lab Scale Permeate Flow Schematic
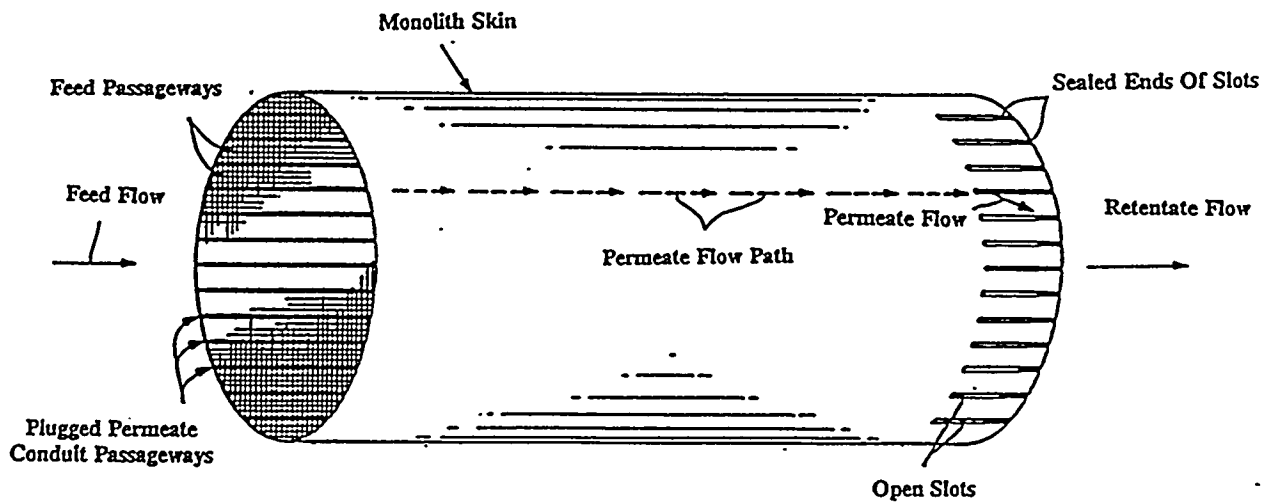
Figure 4.   Full Size Permeate Flow Schematic with Permeate Conduits

the monolith, the monolith is coated with membrane. During operation, feed is pumped through the module, and permeate flows through the membrane into the monolith cell walls. The permeate from any cell in the monolith flows no more than a few cell layers before it arrives at a low pressure permeate conduit. When the conduit is reached, the permeate flow turns toward the end of the monolith containing the slots. Upon reaching the slots, the permeate flow turns 90° and flows into a permeate collection zone.

This approach to removing permeate from the inside of a large diameter monolith results in high surface area modules with very high membrane packing densities. As a result, several advantages are derived from this unique membrane construction. First, since most of the cost of manufacturing ceramic membranes is labor, the cost of producing high surface area membrane elements is relatively low, allowing CeraMem to sell membranes at much lower prices per square foot than other ceramic membranes and at prices competitive to polymeric membranes in some cases. Also, with the high surface area filter elements, fewer elements are needed in any one system, thereby minimizing the amount of associated hardware including housing and seals.

At present, the conduit configuration described above has been commercialized for crossflow liquid applications, using elastomeric boot seals in stainless steel housings, at temperatures up to 200°C. This design, however, is not readily amenable to sealing at higher temperatures due to the temperature limitation on the elastomer boot seal. CeraMem has developed high temperature membrane modules for use in petroleum-based feed stream processing at temperatures up to 350°C on a developmental basis and believes that commercial, full size filter elements with housings and seals capable of operating at higher temperatures are possible.

II. TECHNICAL APPROACH

The primary goal of this program was to develop and demonstrate new ceramic membranes to deash coal liquid bottoms using a two step process. The membrane modules are completely ceramic and are suitable for elevated temperature, hydrocarbon service. The modules have a high packing density and a low production cost. Further, the membrane module structure has an open, "hydrodynamically clean" feed flow configuration and will not be susceptible to plugging by particulates. The first step is ash concentration with crossflow MF. Residual oil, containing ash, is pumped through a ceramic membrane system operating with a transmembrane pressure of about 20-80 psi. If the feed material is generated at ambient pressure, the filtrate will be recovered at ambient pressure. If the feed is pressurized to retain volatiles, then both the concentrate and the filtrate from the membrane unit will be pressurized. The system pump provides the required crossflow velocity through the membrane elements. This concentration process produces two product streams. The filtrate, free of suspended particulates, may be further processed through the liquefaction reactors to increase product yield. The second stream is the solids containing concentrate. The suspended particulates level in the concentrate can be expected to range from about 20 to 30%. If the starting mixture contains 10% solids, concentrating the bottoms to this solids level results in 55-76% recovery of the

liquid fraction as filtrate. Additional processing of the concentrate is necessary in order to extract most of the remaining residual oil.

To increase overall oil recovery, the concentrate can be treated by diafiltration. In this process, shown schematically in Figure 5, a volatile solvent (e.g., a distillate product generated within the coal liquefaction process) is added to the concentrate, and the diluted concentrate is further processed by crossflow MF.

Diafiltration serves to displace the resid from the concentrate with solvent. Therefore, the final concentrate from the diafiltration section consists primarily of suspended particulates and solvent. While diafiltration could be used to recover essentially all of the resid from the ash-reject stream, there will be an economic optimum for the degree of diafiltration actually employed. The diafiltration filtrate is flashed or distilled to recover the solvent, and the diafiltration concentrate is dried to recover solvent.

Experimental Materials and Procedures

Two types of ceramic membranes were tested during this program for coal liquids filtration. The lab scale membrane modules were approximately 12 inches long and 1 inch in diameter and had 1.5 ft^2 of membrane area. The passageways were square and approximately 0.07 inches on a side. The two membranes tested had separation layers consisting of 0.05 μm diameter pore size titania and 0.01 μm diameter pore size silica. Ceramic end rings were bonded onto each end of the module so that it could be sealed into stainless steel housings. The seal between the housing and module was a graphite packing seal used successfully in previous hydrocarbon testing.

The coal liquid and diluent used in these tests were obtained from Hydrocarbon Research, Inc. (HRI) in Princeton, NJ. The coal liquid was a reactor liquid flash vessel bottoms (O-43) from a recent HRI liquefaction run (Run Number 260-004-49-T). The diluent was petroleum-based, hydrotreated startup oil (HRI Number L-809).

The process tests were performed at Imperial Oil, Ltd. in Sarnia, Ontario, Canada. Imperial Oil had a high temperature crossflow test system designed for liquid hydrocarbon testing. The test system was capable of heating feeds to temperatures of about 300°C, at feed stream crossflows of up to 6 gpm and membrane inlet pressures of up to about 100 psig. The system could process the liquids in both recycle mode and batch concentration mode. In recycle mode, the permeate was recycled back to the feed tank resulting in no change in solids concentration in the feed stream. In the batch concentration mode, the permeate was diverted to an alternate vessel resulting in an increase in solids in the feed material.

Two general sets of tests were conducted on one batch of coal liquid. The flash drum bottoms were diluted from approximately 15% total suspended solids to about 10% solids. Solids concentrations were determined by a THF insolubles test, according to a procedure obtained from Consol, Inc. First, filtration tests at constant solids concentration were
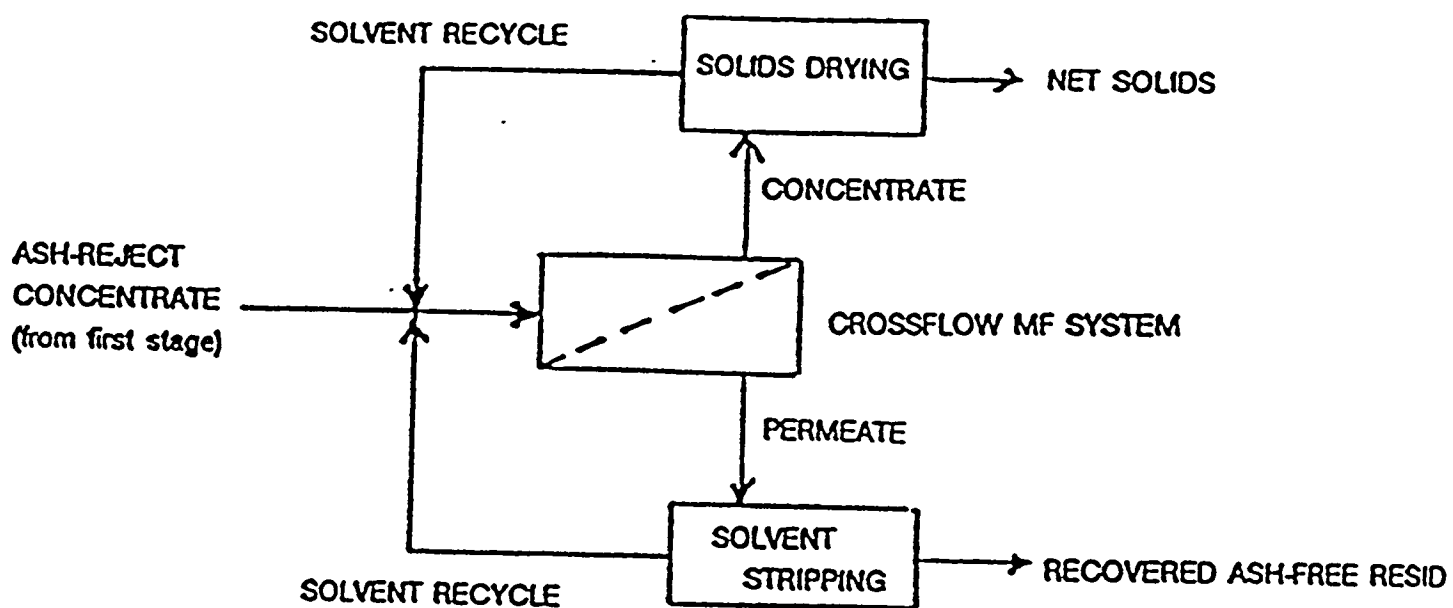
Figure 5.   Proposed Deashing Process Using Microfiltration and Diafiltration

performed to determine the effects of membrane type, temperature, pressure, and crossflow velocity. Based on the results of these initial tests, the membrane type and process conditions for the batch concentration of the coal liquid to about 20% solids was determined. After this initial batch concentration was performed on the diluted flash drum bottoms, four additional dilutions and concentrations were performed at the same process conditions. Samples of feed and permeate from each cycle were analyzed for THF insoluble solids. These results, together with the masses of liquid added to or sampled from the feed tank or permeate stream, were used to estimate the amount of residual oil left in the concentrate at the end of each cycle. Due to the very similar boiling point curves of the petroleum-based startup oil and the coal derived liquid, distillation could not be used to directly determine the concentration of residual oil in the concentrate samples.

III. EXPERIMENTAL RESULTS

A. Testing of the Ceramic Membrane

Initial process flux characterization experiments were conducted with the two different membrane module types using the diluted coal liquid bottoms. The feed was charged into the system along with one of the membrane modules, and the feed was heated in recycle over the course of two days to 265°C. Permeate flux was then measured over the course of several hours at 265°C, 240°C, and 200°C. Transmembrane pressure was 80 psig and the crossflow was about 6 gallons per minute (gpm). Permeate samples were analyzed for non-THF soluble solids. The same process was repeated for the second membrane type. The data for both membrane types are included in Table 1.

Several clear observations can be made concerning the data. First, the 0.01 µm and 0.05 µm membranes were very different in terms of process flux. The 0.05 µm membrane had a very good crossflow process flux of over 200 kg/m^2/h which was a factor of twenty higher than the 0.01 µm membrane. Second, the process flux appeared to be strongly dependent on temperature between 200°C and 265°C. Third, non-THF soluble solids retention was very high for both types of membranes.

Based on the membrane evaluation tests, the 0.05 µm pore size titania membrane was selected for further testing to evaluate the effects of various process parameters on membrane flux performance. The purpose of these parametric tests was to determine the process conditions for subsequent concentration/diafiltration process runs.

Table 1.
Ceramic Membrane Performance in Deashing of Coal Liquids

Evaluation	0.01 µm Silica		0.05 µm Titania	
	Temperature (C)	Flux (kg/m^2-h)	Temperature (C)	Flux (kg/m^2-h)
Process Flux	265	11	265	223
	240	5	240	198
	200	1.3	200	98
Solids Retention	> 99.9%		> 99.7%	

Process Variable Evaluation

The data recorded during the process variable evaluation experiments is shown in Table 2. The permeate flux level increased by 17% with an increase in temperature from 200°C to 270°C. This increase appears to be small compared to the anticipated increase based on the data obtained earlier in the project (Table 1). Data obtained on a 0.05 µm pore size titania membrane showed an increase of about 120% from 200°C to 265°C. However, the flux performance at about 265°C in both cases was almost the same, 220-225 kg/m²-h. The cause for this observation is probably that the first data were obtained by heating the feed directly to 265°C, then measuring flux performance as the temperature decreased over the course of about 5 hours. In the parametric tests, the 200°C data were taken first and then the feed was heated to 269°C overnight before the flux data at high temperature was taken. If membrane fouling is occurring over time, then the effect of temperature will appear to be enhanced in the first case. Both membrane fouling and temperature decrease will decrease flux, thereby making the low temperature flux in the first set of data look very low. In the second case, the high temperature data will have been taken after a longer processing time, and the flux rate will be decreased due to membrane fouling. Consequently, the increase in flux due to increased temperature in the parametric test was tempered by time effects possibly as a result of adsorption of materials onto the titania membrane during processing.

The effect of time is more clearly shown in Table 2 by comparing the data taken at 22 and 27 hours. With a very small reduction in temperature, the flux falls off by about 45%. It appears that membrane fouling, possibly by adsorption of material onto the membrane or plugging of the pores by very fine colloidal material, is occurring. The effect of membrane fouling is to reduce permeate flux as flux inhibiting material (foulants) is brought to the. membrane via permeation flow. As long as there are foulants in the feed material and they can attach to or plug the membrane, flux will decrease. Membrane modifications or process changes (e.g., feed pretreatment) can have a significant impact on the degree of fouling and should be studied before commercial systems are designed. While membrane

fouling is an important process characteristic, it does limit the usefulness of the parametric studies.

Table 2
Coal Liquid Deashing Parametric Studies Using Titania Membrane

Elapsed Time (hrs)	Crossflow (gpm)	Temperature (Deg C)	Average Pressure (psi)	Differential Pressure (psi)	Permeate Flux (kg/m^2-h)
1.1	5.9	195	77.5	3	193.1
Filter element and feed material slowly heated overnight with no permeate flow.					
22.1	5.8	269	74.0	8	225.7
27.0	6	254	76.0	8	122.8
Transmembrane pressure decreased after 27 hours and allowed to stabilize for four hours.					
31.1	5.9	254	37.5	9	76.8
46.5	6	254	39.0	4	80.2
51.5	5.8	254	38.5	3	67.0
Crossflow decreased after 51.5 hours and allowed to stabilize for 2.5 hours.					
54.3	4	243	39.5	3	56.4
70.7	4	243	37.5	3	45.7

The next process variable evaluated was pressure across the membrane. It can be seen in Table 2 that the permeate flux fell off about 40% with a 50% reduction in the pressure. A large decrease in flux level with reduced membrane driving force is typical for microfiltration applications. However, it is unclear how much membrane fouling contributed to this effect. The relative stability of the flux level over the next 20 hours indicated that the rate of membrane fouling was decreasing. This may occur due to membrane conditioning (i.e., reduction in adsorption sites) and/or a reduction in the amount of foulant in the feed material. Since the experiment was run with a single batch of material, the foulant could have been stripped out of the fluid and onto the membrane. The rate of membrane fouling would decrease as the amount of foulant remaining in the feed decreased.

Lastly, the effect of crossflow rate on membrane flux performance was investigated. The effect of crossflow rate in some applications can be large due to the sweeping action of the fluid across the membrane removing flux inhibiting materials from the membrane surface. As can be seen in Table 1, a 33% decrease in crossflow caused a 16% decrease in flux. Once again, membrane fouling may have affected the observed results.

The process conditions chosen for the concentration/diafiltration runs were those that maximized process flux in the parametric tests. These conditions were 270°C, 80 psig inlet pressure, and 6 gpm crossflow rate.

Diafiltration Process Evaluation

Data from the concentration/diafiltration runs is summarized in Figures 6, 7, and 8 and Table 3. Raw data from these runs is presented in the appendix. Process flux data from concentration Runs 1, 3, and 5 are shown in Figures 6, 7, and 8, respectively. The data are presented using a linear trend line through the flux data points. The trend line is used to give a general guide to the fall off in flux during concentration and is not curve fit to the data which would better indicate the function for flux fall off. The flux data indicate that the concentration run fluxes were fairly similar to each other. This may have been due to the use of start-up oil which is similar to the liquid in the atmospheric bottoms. Use of a lower molecular weight solvent that would need to be pressurized at process temperature but would allow for relatively easy removal from the concentrate and permeate by flashing would probably give different results. The overall flux rate is very encouraging. Flux levels for typical crossflow applications range from 20 to 200 l/m²-h. Considering the high viscosities and solids concentrations in these streams, the flux levels are very good. A high flux rate will have a large impact on reducing the number of filters required in a commercial system.

Data in Table 3 include the measured solids concentration of feed and concentrate samples for each concentration run as well as the calculated amount of residual coal-derived oil in each sample. As can be seen, the amount of concentration that can be obtained with these membrane filters under these process conditions is about 20% solids. Over the course of these concentration runs, most of the residual oil has been extracted from the bottoms. It was calculated that 23% of the oil was removed from the bottoms after 3 dilution/concentration runs, while 7% of the residual oil was removed from the bottoms after 5 dilution/concentration runs. This data will be helpful in evaluating the process design and process economics.

Table 3.
THF Insoluble Solids and Residual Oil Concentrations of Each Process Cycle

	Concentration Runs				
Property	Number 1	Number 2	Number 3	Number 4	Number 5
Feed Solids Conc.	7.2%	9.4%	8.7%	7.8%	7.8%
Retentate Solids Conc.	29.4%	22.1%	17.1%	16.5%	18.8%
Retentate* Residual Oil Conc. (Calc)	62.1%	40.3%	22.8%	11.3%	6.8%

* Residual oil concentration in concentrate calculated on a diluent-free basis. This assumes that in an actual process the diluent would be flashed off, leaving only solids and residual oil.
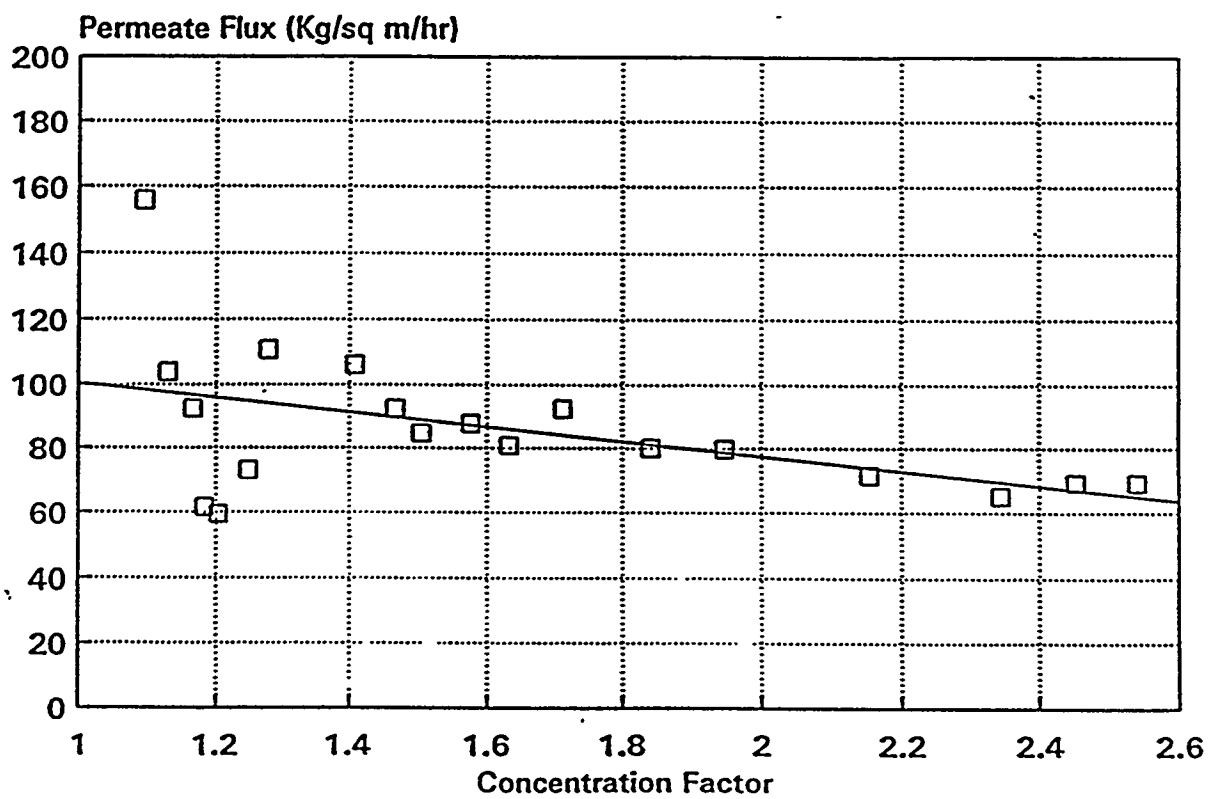
Figure 6.   Coal Liquid Process Flux Versus Concentration in Run 1

Permeate Flux (Kg/sq m/hr)
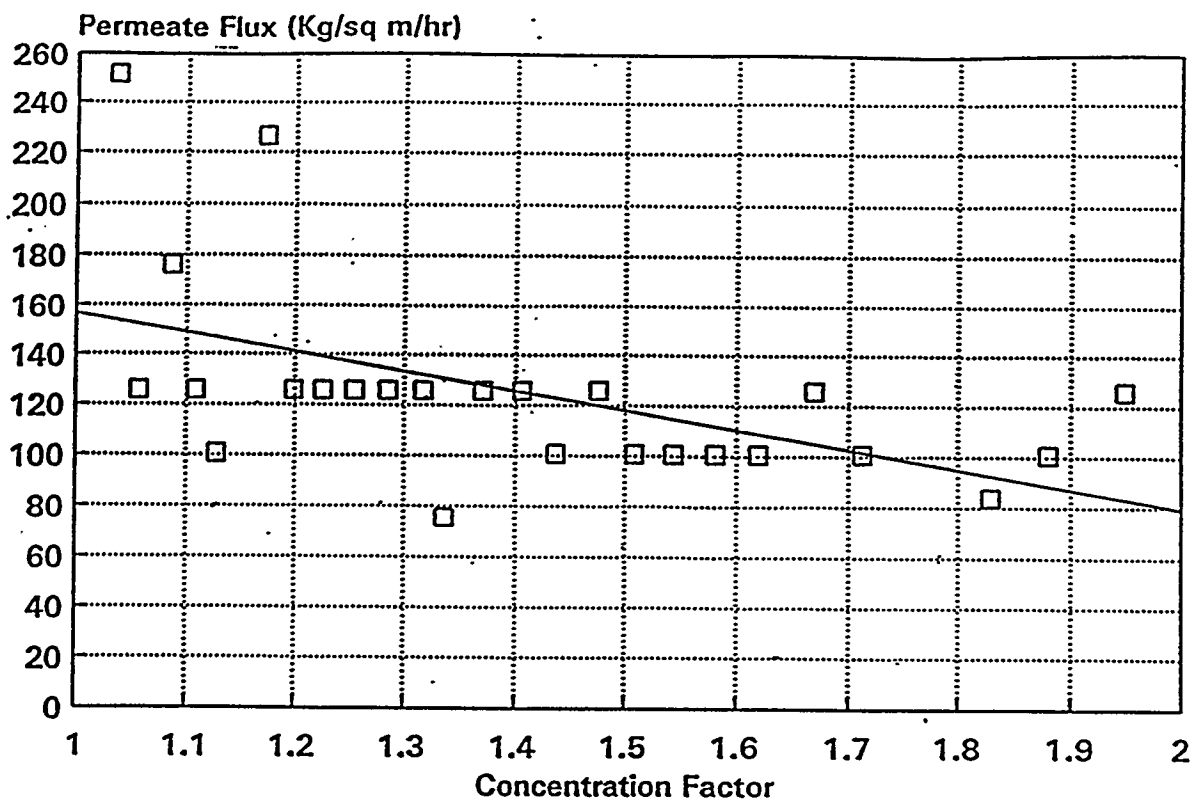
Figure 7.　　Coal Liquid Process Flux Versus Concentration in Run 3

Permeate Flux (Kg/sq m/hr)
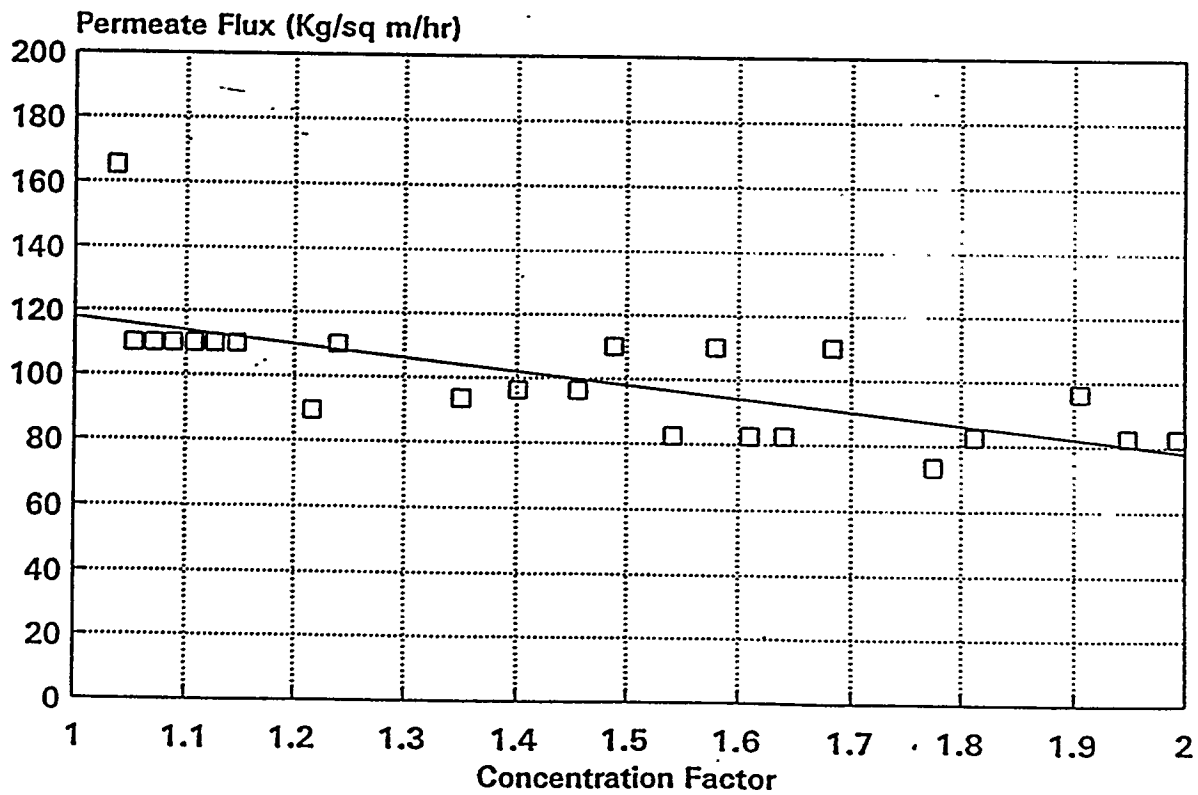
Figure 8　　Coal Liquid Process Flux Versus Concentration in Run 5

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Analysis of Samples from Batch Concentration Trials

CeraMem worked with Consol, Inc. on the analyses of initial feed, permeate and concentrate samples from each of the diafiltration/concentration runs. The goal was to develop test methods to analyze the samples in order to determine the quantities of starting coal liquid (O-13 reactor flash drum bottoms) and diluent (HRI's petroleum-based hydrotreated startup oil) in the process concentrates and permeates. The concentration of coal liquid in the concentrates and permeates is an important factor in analyzing the diafiltration/concentration process used in the project. The data would be used to confirm the calculated amounts of coal liquid and diluent in each of the process streams assuming the two liquids were miscible. Due to the similarities of the coal liquids and the diluent, an analytical procedure could not be developed to differentiate between them. These efforts are discussed in the appendix as Attachment 7.

Membrane Cleaning Procedures

Preliminary work was also performed under this program to test membrane cleaning procedures required for long term, continuous operation in a coal liquefaction plant. The module used in concentration Runs 2 to 5 (AD-2053) of the diafiltration/concentration process tests had 12 plugged channels following the series of tests. After this module was soaked in toluene overnight at room temperature, it still contained 12 plugged channels. A toluene flux was performed on the module in the same manner that the initial toluene flux was measured. Solvent flux was measured in a separate system, not modified for coal liquids operation, at room temperature using toluene at 13 psig transmembrane pressure. The toluene flux after this simple soak cleaning was 18 kg/m^2-h, which was down from 425 kg/m^2-h initially. Scaling the toluene flux to the process transmembrane pressure gives a flux of about 115 kg/m^2-h which is very similar to the process fluxes obtained. Basically, the toluene soak did not clean the module. In addition, module AD-2076 which was the 0.01 μm silica membrane used in the membrane evaluation experiments was soaked in room temperature toluene for three days, after which the toluene flux was measured. No flux was obtained from the module. This is not surprising in that the coal liquid had been sitting inside the module for several months before soaking in toluene. In subsequent efforts it would be desirable to test hot solvent and alternative solvents for removal of a significant amount of solidified material from the membrane.

B. Preliminary Design Approach and Process Economics

CeraMem has conducted an investigation of new crossflow ceramic membranes and the processes necessary to deash and extract coal-derived oil from coal liquids bottoms. Ideally, results from the experimental tasks would be used to develop a preliminary engineering design and cost estimate for a demonstration pilot system incorporating full scale membrane modules. This would also include the development of test protocols for on-site evaluations.

Another task under this program was to develop first estimates for microfiltration capital and operating costs and assess process economic feasibility. Membrane flux measurements from both batch concentration and differential trials were used to begin sizing a membrane system of commercial capacity. Estimates of both installed plant costs and operating costs were developed.

The basic system design that could be used in a commercial scale direct coal liquefaction plant is shown in Figure 9. In the proposed process, the residual oil from coal liquids is separated by "washing" with a volatile solvent in a ceramic membrane process (ultrafiltration). This washing process is widely known in the membrane industry as "diafiltration." The coal liquid resid can be either from the atmospheric still or the vacuum still (as discussed further below). The two feeds shown in Figure 10 for the CTSL Process have been considered in this economic study. These have insoluble contents (ash and unreacted carbon) of about 9% and 18%, respectively.

For the diafiltration process with atmospheric resid, the process considered entails an initial ceramic membrane "concentration" step, in which the atmospheric resid is concentrated by ultrafiltration to 18% insoluble matter. That is, during this step about one-half of the resid contained in the atmospheric bottoms is deashed in a membrane process prior to a diafiltration operation.

For the diafiltration process with the vacuum resid, no preconcentration is used, and diafiltration is conducted directly with the bottoms stream.

In the diafiltration process (with or without the preconcentration step), the resid is admixed with a volatile distillate solvent from the atmospheric column. The important properties of this solvent are that it be fully miscible with the resid and that it be readily separated from the resid by a flash or distillation process.

As shown in Figure 9, the permeate (the stream passing through the membranes) is fractionated in a still. The solvent recovered is recycled to the front end of the diafiltration process. The bottoms is the deashed resid product. The concentrate (or retentate) from the diafiltration process is also fractionated. The bottoms stream is highly ash-enriched and is either recycled to the coal liquefaction process, burned for fuel, or used for hydrogen production.

The diafiltration process schemes would replace a series of steps in an existing resid processing scheme as follows:

Atmospheric Bottoms Process: Replaces the Vacuum Still and ROSE-SR

Vacuum Bottoms Process: Replaces ROSE-SR
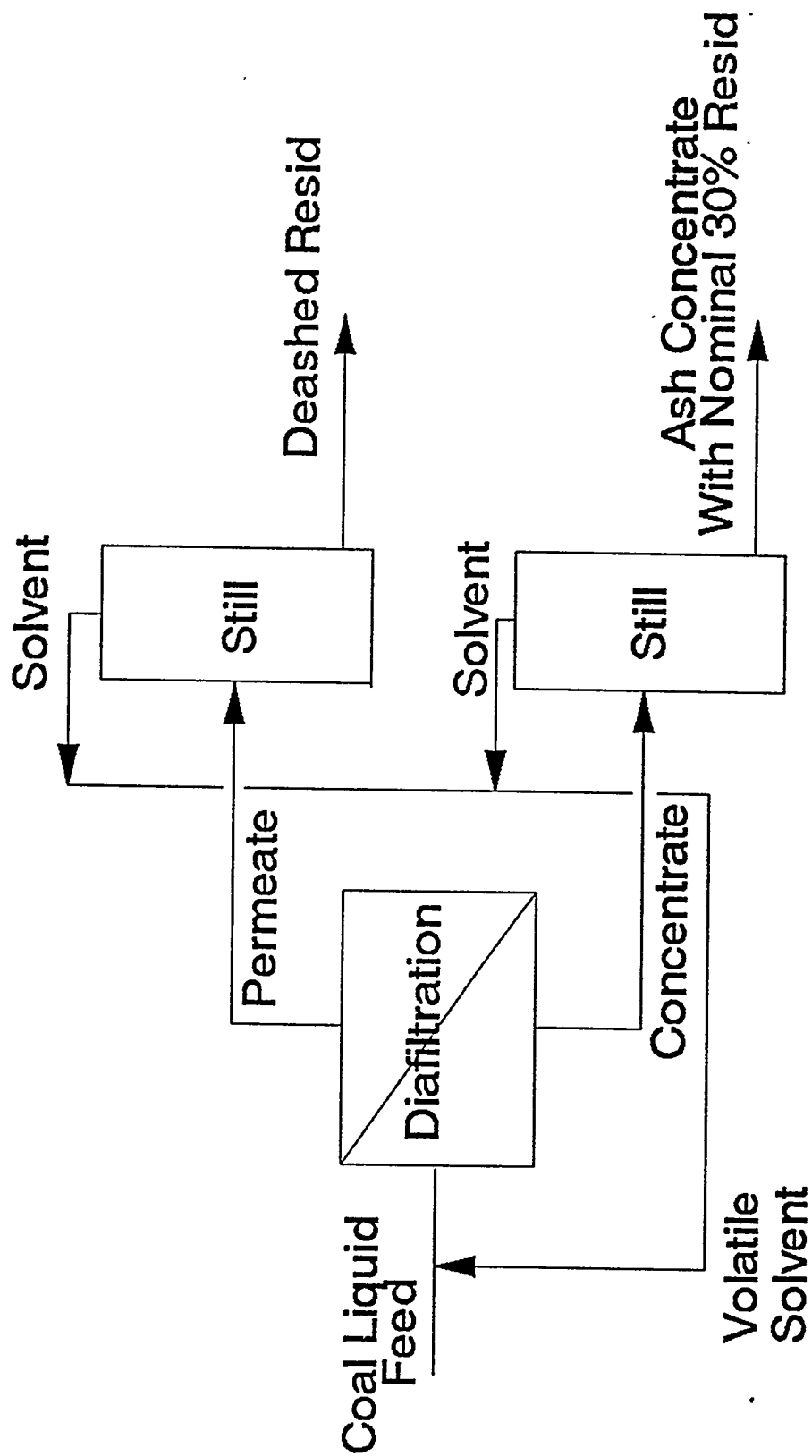
Figure 9. Block Diagram For Coal Liquids Diafiltration

Figure 10. CTSL Process (Illinois # 6 Coal) Based on HTI Run CMSL-03
Based on 100# MAF Coal Feed
Scale Factor for Commercial Plant = 12,600
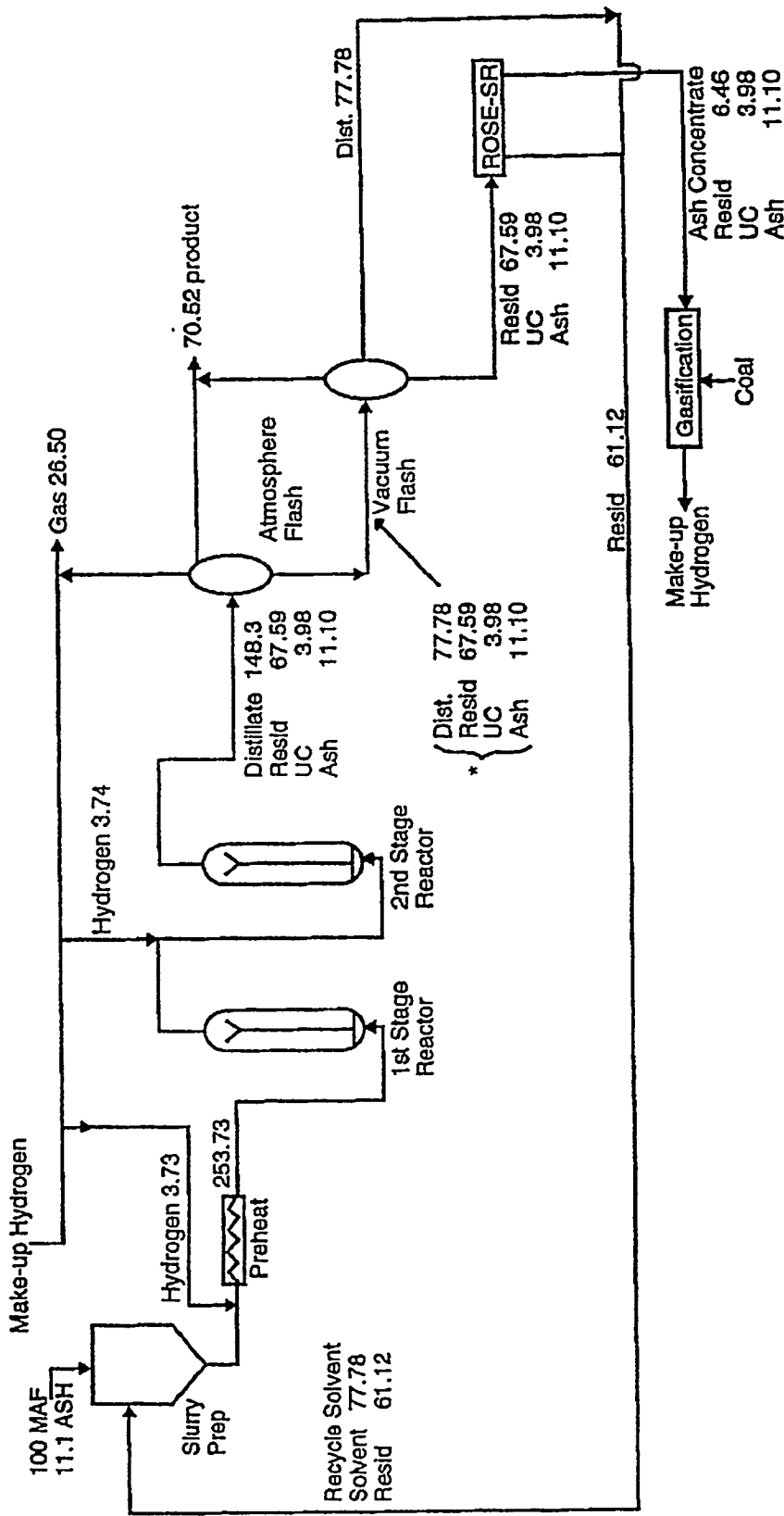
Based on Wilsonville Run 257E, Illinois #6

*650°F-	15.8
650-850°F	65.0
850-975°F	21.0
975°F+	43.5
UC	3.98
Ash	11.10

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There are three principal means by which diafiltration can be practiced: two are continuous processes, and one is a batch process. These are shown schematically in Figures 11-13. The processes are identified in the following text as (a) continuous, stages-in-series with "parallel" diafiltration; (b) continuous, stages-in-series with "counter-current" diafiltration; and (c) "batch." The batch process is substantially more efficient, as will be demonstrated below.

In general, the batch process is the most efficient, both in minimizing membrane area (and plant costs) as well as minimizing the volume of diafiltration solvent employed in the washing step. The latter is important in that it determines the size and cost of the permeate still, as well as the energy consumed in the permeate still.

Six design cases have been considered which incorporate the new process.

1. Four Stage in Series Diafiltration, Vacuum Resid Feed, Counter-Current Diafiltration
2. Four Stages in Series Diafiltration, Vacuum Resid Feed, Parallel Diafiltration
3. Four Stages in Series Diafiltration, Atmospheric Resid Feed, Counter-Current Diafiltration
4. Four Stages in Series Diafiltration, Atmospheric Resid Feed, Parallel Diafiltration
5. Batch Diafiltration, Vacuum Resid Feed
6. Batch Diafiltration, Atmospheric Resid Feed

There are two major differences between these cases. In using the vacuum bottoms, there is less fluid that has to be processed, and the bottoms can be diluted in the first diafiltration stage. Using the atmospheric bottoms as the feed material requires an extra stage of membranes to perform the initial concentration but also would eliminate the need for the vacuum still.

Second, the diafiltration can be performed in parallel with the fluid processing or in counter-current mode. In the parallel mode, fresh solvent is injected into each of the membrane stages thereby obtaining the most oil extraction but using large quantities of solvent. In counter-current mode, fresh solvent is injected into the last stage only and using solvent-containing permeate in each of the previous stages. This mode minimizes the use of solvent in the diafiltration process but requires many more membrane elements.

The system would have four stages of membranes each containing approximately the same number of membrane elements. Each stage of membrane elements has its own recirculation pump to create the crossflow necessary to sweep the membrane surface clean. Pressure to drive the permeate through each of the membranes of the four stages is generated by a feed pump which feeds new material into the first stage. The solids-free permeate is collected in a common piping system and recycled for further processing. The retentates of each of the stages are sent as feed into the following stages. The liquid is diluted in the recirculation loop with diafiltration solvent. In this way, the coal-derived oil is continuously extracted from the solids in the bottoms. The final retentate (concentrate)
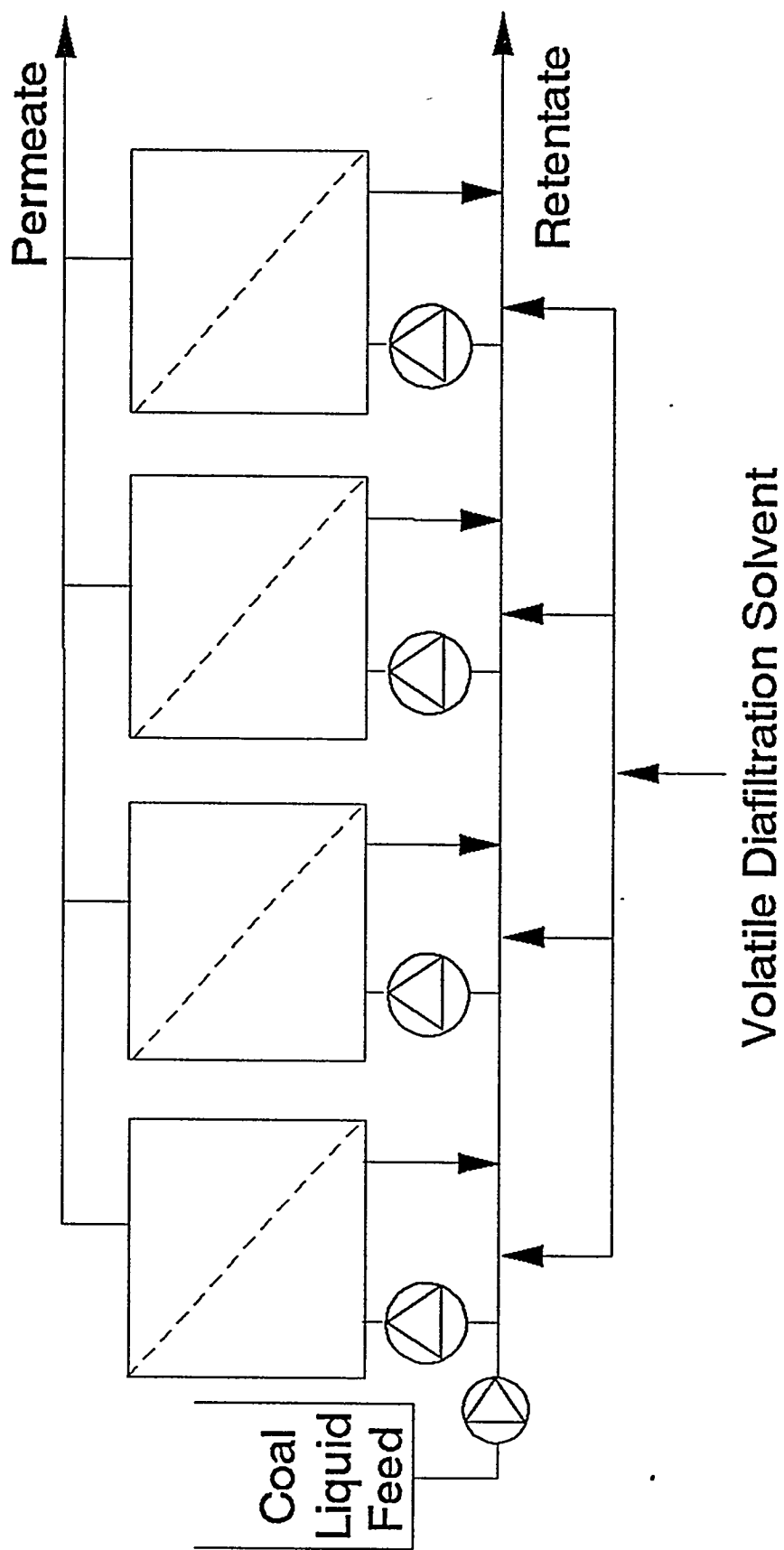
Figure 11. Four Stage Diafiltration System
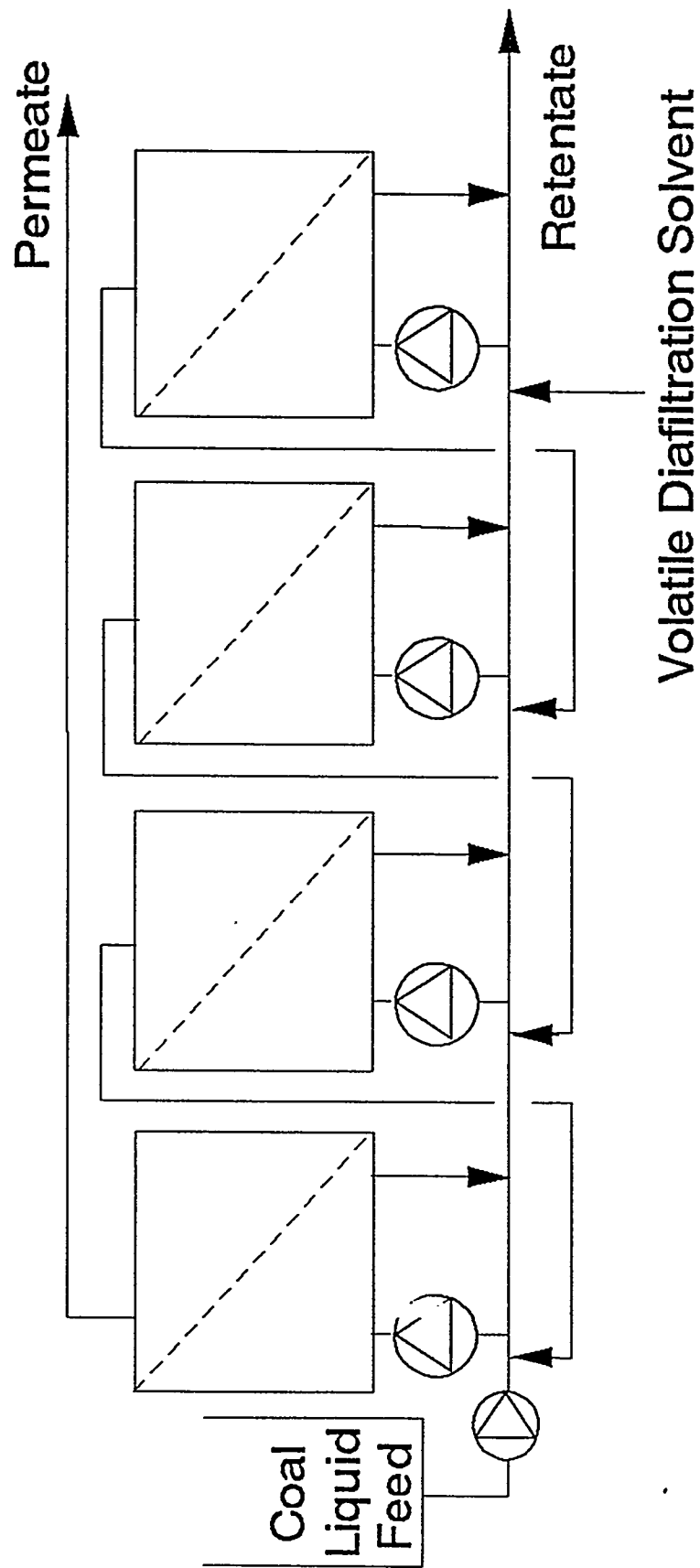
Figure 12. Four Stage Counter-Current Diafiltration Process
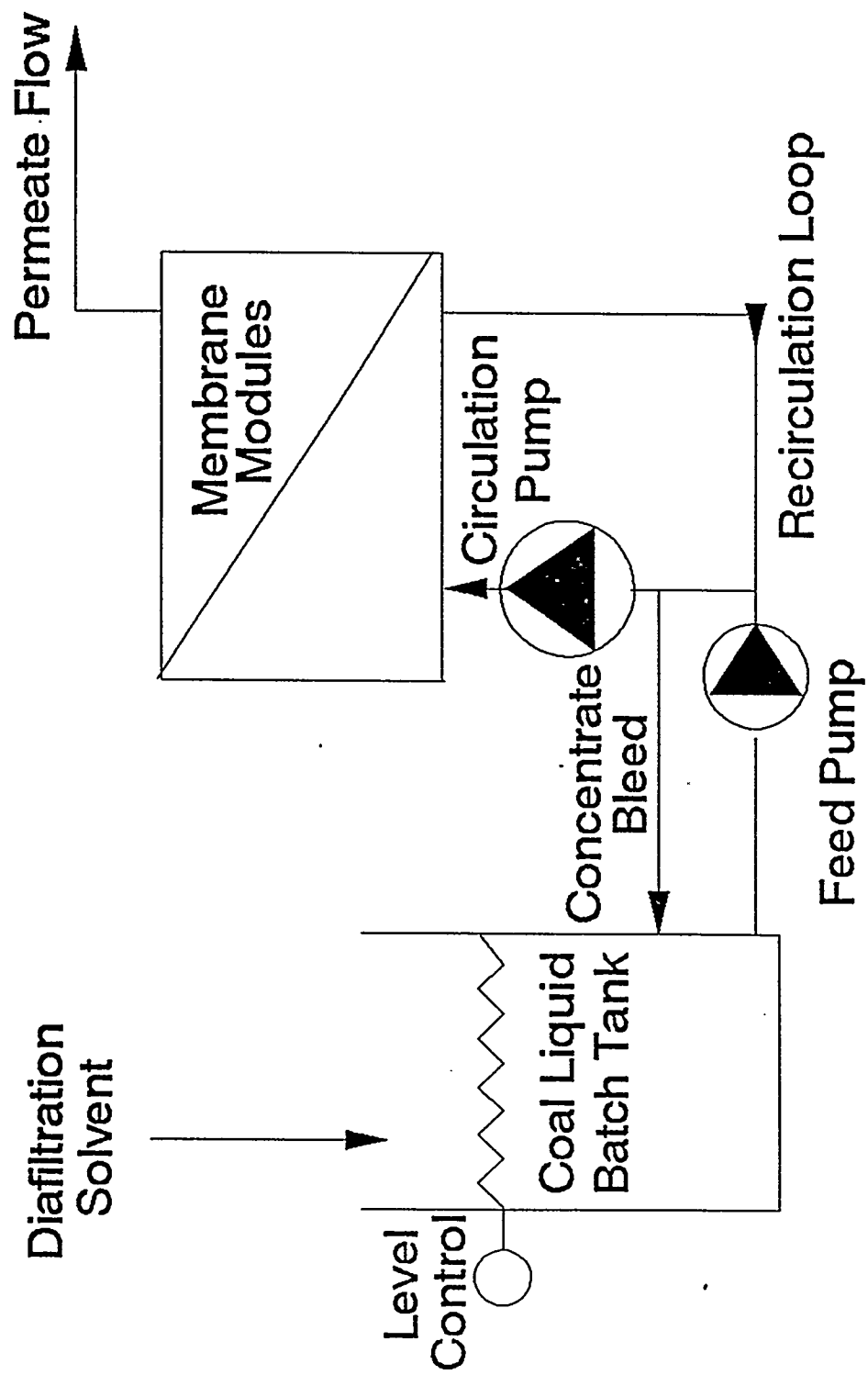
Figure 13. Batch Diafiltration System

·solids can be sent to a gasifier after flashing off the diafiltration solvent which will be subsequently recycled within the process.

The cases are all based on a plant processing 15,000 tons of coal per day, with an atmospheric still bottoms flow of 820,000 kg/h and a vacuum still bottoms flow of 423,000 kg/h. The design bases, process conditions, and the results of the analysis are given in Table 4. Table 4 also gives the membrane areas and number of membrane modules anticipated to be required (each with 11 m^2 membrane area). Also, membrane plant costs (+/- 25%) are given for the six options. The membrane plant costs are those for similar systems developed for explosion proof membrane systems for processing solvent containing latex suspensions. Table 5 gives the estimated membrane replacement costs and operating cost information (+/- 25%). The costs, expressed both in terms of number of membrane modules and per unit membrane area, are given in Figures 14 and 15.

Table 4.
Estimated Purchased System and Operating Costs
Coal Liquids MF Plant

Plant Size:

tons coal/day	Bbl/day	Coal Liquids kg/hr	Atm. Bottoms kg/hr	Vac. Bottoms kg/hr
15,000	75,000	820,000	423,000	423,000

Bases For Design Cases:

Case	Operating Mode	Bottoms Feed	Insoluble Solids	Flow, kg/hr	Preconcentration	Diafiltration Mode	Retentate-Resid % (Solvent-free)
1	Stages-In-Series	Vacuum	18	423,000	no	Counter-Current	30
2	Stages-In-Series	Vacuum	18	423,000	no	Parallel	30
3	Stages-In-Series	Atmospheric	9	820,000	yes (2X)	Counter-Current	30
4	Stages-In-Series	Atmospheric	9	820,000	yes (2X)	Parallel	30
5	Batch	Vacuum	18	423,000	no	Batch	30
6	Batch	Atmospheric	9	820,000	yes (2X)	Batch	30

Process Flow Information & Estimated Purchased System Costs:

Case	Operating Mode	Bottoms Feed	DF Solvent Flow, kg/hr	Feed Flow/ Membrane Area kg/hr-sq m	Membrane Area, sq ft	Number of Membrane Modules	Estimated System Price
1	Stages-In-Series	Vacuum	476,000	20	252,000	2,100	$22,000,000
2	Stages-In-Series	Vacuum	1,015,000	37.5	134,400	1,120	$15,000,000
3	Stages-In-Series	Atmospheric	459,000	32.7	300,000	2,500	$26,000,000
4	Stages-In-Series	Atmospheric	984,000	53	184,200	1,535	$18,000,000
5	Batch	Vacuum	425,000	51.5	90,000	750	$10,000,000
6	Batch	Atmospheric	425,000	64.2	139,200	1,160	$15,000,000

Note: Feed flow/membrane area determined from process design calculations. These include material balance calculations and an assumed membrane flux of 90 liters/square meter-hour.

Table 5.
Estimated Purchased System and Operating Costs
Coal Liquids MF Plant

Estimated Membrane Replacement and Power Costs

Case	Operating Mode	Number of Modules	Membrane Area, sq ft	Module Life, yrs	Module Cost, $/sq ft	Module Costs, $/yr
1	Stages–In–Series	2,100	252,000	2	20	$2,520,000
2	Stages–In–Series	1,120	134,400	2	20	$1,344,000
3	Stages–In–Series	2,500	300,000	2	20	$3,000,000
4	Stages–In–Series	1,535	184,200	2	20	$1,842,000
5	Batch	750	90,000	2	20	$900,000
6	Batch	1,160	139,200	2	20	$1,392,000

Case	Operating Mode	Number of Modules	Module Module, kw	Total Power Kilowatts	Power Costs $/kw–hr	Power Costs $/year
1	Stages–In–Series	2,100	2	4,200	$0.05	$1,764,000
2	Stages–In–Series	1,120	2	2,240	$0.05	$940,800
3	Stages–In–Series	2,500	2	5,000	$0.05	$2,100,000
4	Stages–In–Series	1,535	2	3,070	$0.05	$1,289,400
5	Batch	750	2	1,500	$0.05	$630,000
6	Batch	1,160	2	2,320	$0.05	$974,400

Case	Operating Mode	Total Costs $/year	Combined Costs Costs, $/bbl
1	Stages–In–Series	$4,284,000	0.16
2	Stages–In–Series	$2,284,800	0.09
3	Stages–In–Series	$5,100,000	0.19
4	Stages–In–Series	$3,131,400	0.12
5	Batch	$1,530,000	0.06
6	Batch	$2,366,400	0.09
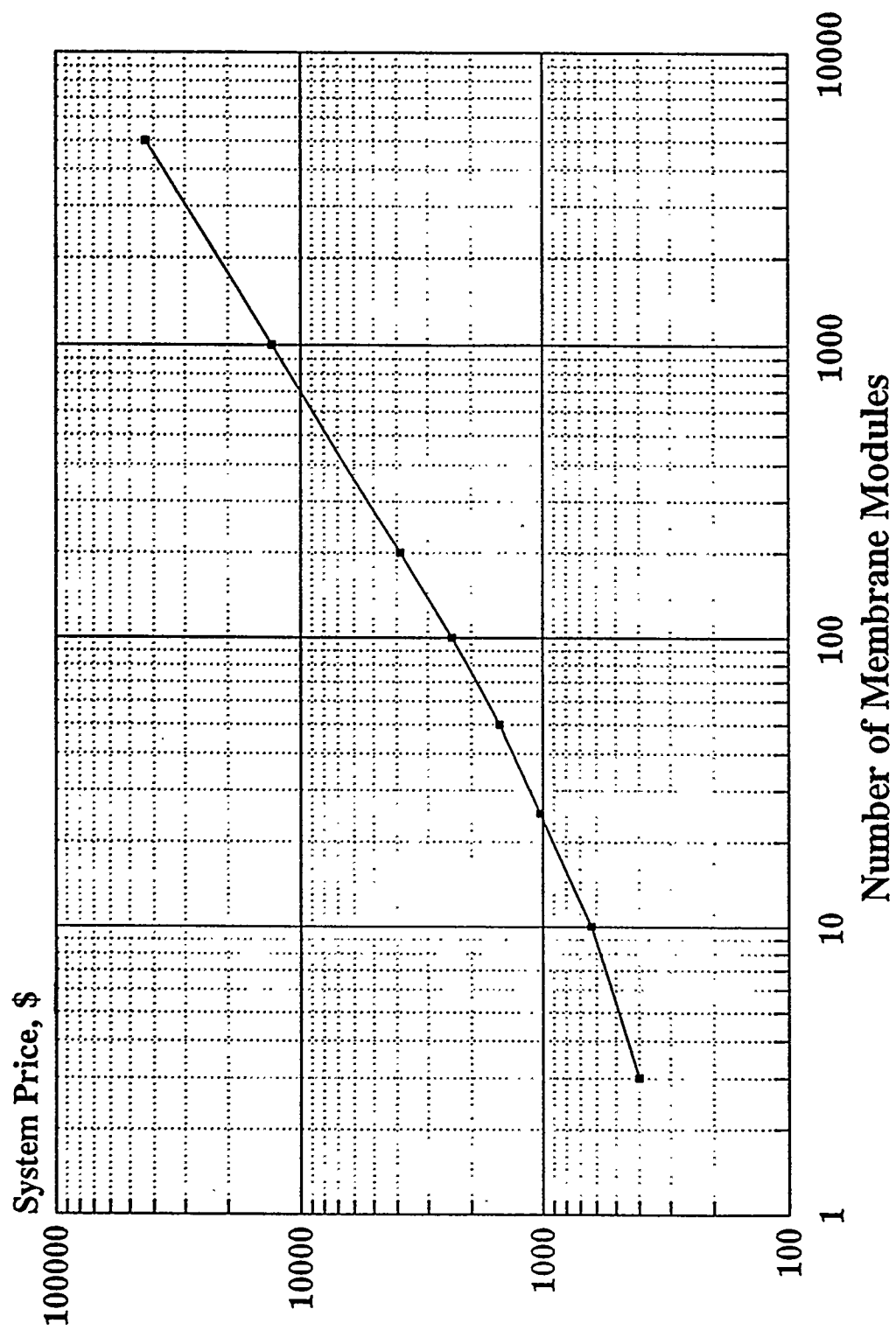
Figure 14. Price Estimates for MF Systems [Stainless Explosion Proof Designs]
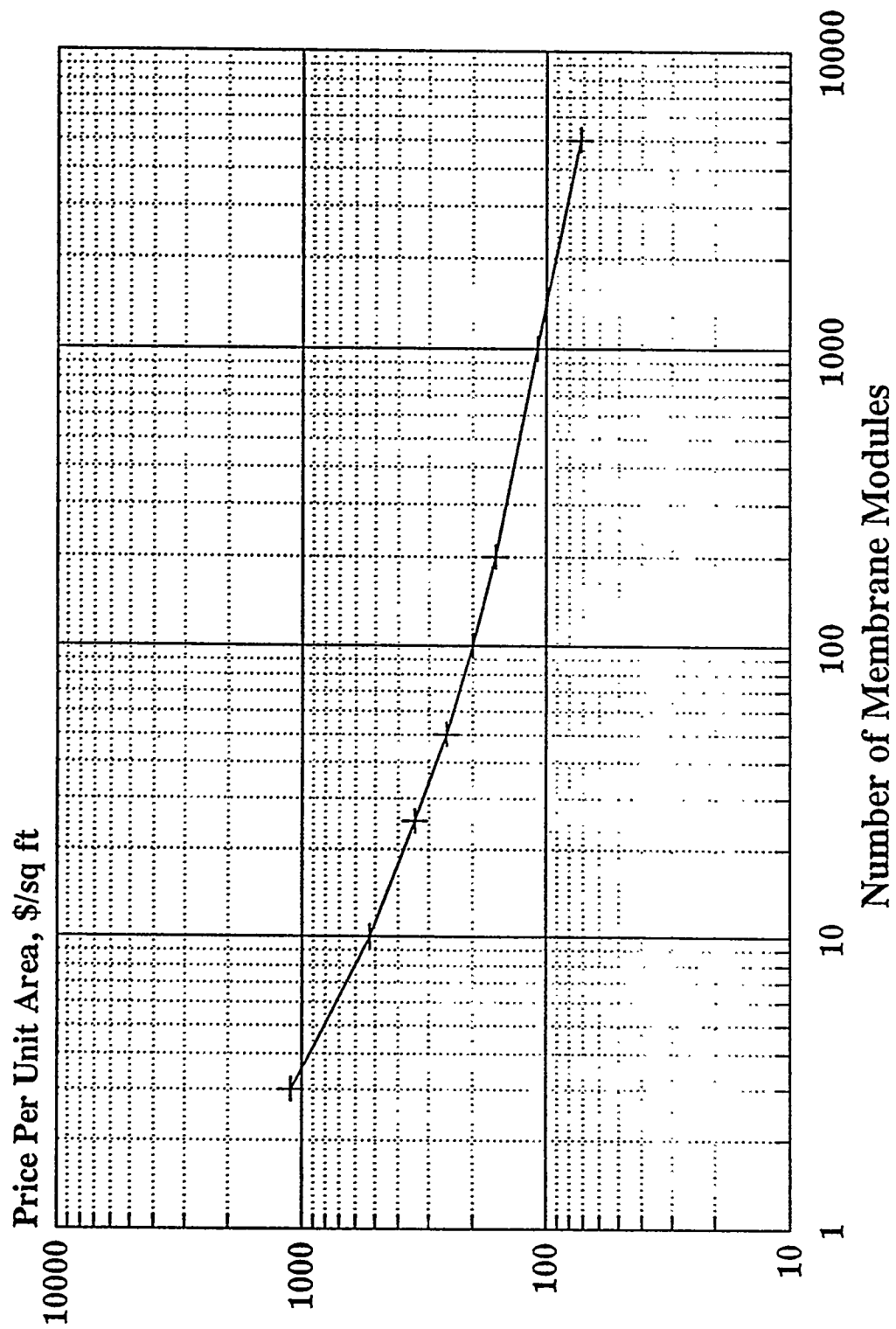
Figure 15. Price Estimates for MF Systems - Price Per Unit Area
[Stainless Explosion Proof Designs]

IV. CONCLUSIONS AND RECOMMENDATIONS

The primary goal of this experimental program was to demonstrate concept feasibility and to establish the specific types of ceramic membranes which are suitable for deashing of coal liquids. Several process variables such as process flux, ash rejection, and membrane cleanability were studied under different operating conditions (temperature, pressure, and crossflow velocity).

Based on work performed to date, CeraMem concludes that 0.05 μm nominal pore size titania membrane crossflow filters can efficiently filter diluted atmospheric flash drum bottoms with excellent solids removal (> 99%) and high fluxes at 270°C (100 to 200 l/m^2-h). In addition, a diafiltration process can be designed around these filters. If a relatively volatile diafiltration solvent can be found that is (1) soluble in the coal liquid and (2) capable of being recycled after flashing it off the permeate and concentrate streams, a potentially cost effective MF membrane diafiltration system can be developed.

The results of this experimental program form the basis for design and fabrication of a demonstration deashing system using full scale membrane modules. Towards this end, CeraMem plans to work with Mitre Corporation on further refining the cost estimates for MF membrane diafiltration systems integrated into a direct coal liquefaction process plant. If the costs look attractive, CeraMem believes that additional research to find appropriate diafiltration solvents and experiments on fresh coal liquids from a direct liquefaction process development unit, such as the one at HRI, would be warranted.

Several specific recommendations are made for a Phase II effort:

- Identify and test alternative diafiltration solvents having volatility (and a distillation curve) different from the particulate free coal liquid product.

- Evaluate these new solvents and their effectiveness upon recycle to the diafiltration system.

- Develop and test more aggressive procedures for membrane cleaning, particularly for removal of large amounts of solidified material, such as the use of hot solvents and alternative solvent types.

- Develop detailed engineering design and cost estimate for a demonstration pilot system incorporating full scale membrane modules.

References

1. Harland, et. al., "Research On Cross-Flow Filtration for solids Removal from Coal Syncrudes," final report for DOE Contract No. EX-78-C-01-2245, NTIS Report FE-2245-16, Feb., 1978.

2. J.Arod, et. al., "Process For The Treatment Of A Hydrocarbon Charge By High Temperature Ultrafiltration," US Patent 4,411,790, 10/25/83.

3. Giuliani, et. al., "Method For The Liquid Phase Hydrotreatment Of Heavy Hydrocarbons In The Presence Of A Dispersed Catalyst," US Patent 4,756,821, 7/12/88.

APPENDIX

Attachment 1:   Data summary from concentration runs.

Attachment 2:   Data table for first concentration run.

Attachment 3:   Data table for second concentration run.

Attachment 4:   Data table for third concentration run.

Attachment 5:   Data table for fourth concentration run.

Attachment 6:   Data table for fifth concentration run.

Attachment 7:   Development of Analytical Tests for Coal Liquids and Solvent

Attachment 1
Data Summary from Concentration Runs

Coal liquid Concentration Run Number 1; 9/19/94

Time	Delta P	Total Permeate	Delta Permeate	Conc Factor	Perm Flux	Feed Temp	Inlet Pres	Feed Flow
0.0	4.0	0.0	0.0	1.0		273.0	78.0	6.0
16.0	5.0	5.4	5.4	1.1	155.8	274.0	78.0	5.8
24.0		7.2	1.8	1.1	103.8			
33.0		9.0	1.8	1.2	92.3			
39.0		9.8	0.8	1.2	61.5			
46.0		10.7	0.9	1.2	59.3			
58.0	6.0	12.6	1.9	1.2	73.1	274.0	79.0	5.8
63.0		13.8	1.2	1.3	110.8			
83.0		18.4	4.6	1.4	106.2			
92.0	4.0	20.2	1.8	1.5	92.3	271.0	78.0	6.0
98.0		21.3	1.1	1.5	84.6			
108.0		23.2	1.9	1.6	87.7			
116.0		24.6	1.4	1.6	80.8			
125.0		26.4	1.8	1.7	92.3			
140.0	4.0	29.0	2.6	1.8	80.0	270.0	81.0	6.0
151.0		30.9	1.9	1.9	79.7			
171.0	8.0	34.0	3.1	2.2	71.5	272.0	80.0	5.7
188.0		36.4	2.4	2.3	65.2			
196.0	12.0	37.6	1.2	2.5	69.2	274.0	81.0	6.0
202.0	13.0	38.5	0.9	2.5	69.2	---	81.0	6.0
Minutes	psi	Kg	Kg	Kg Basis	Kg/sq m/hr	Deg C	psi	USGPM

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Table 1. Coal Liquids Concentration Run Number 1

Coal liquid Concentration Run Number 2; 9/27/94

Time	Delta P	Total Permeate	Delta Permeate	Conc Factor	Perm Flux	Feed Temp	Inlet Pres	Feed Flow
0.0	2.0	1.6	0.0	1.0		271.0	78.0	5.8
6.0	2.0	3.3	1.8	1.1	137.3	271.0	78.0	5.8
13.0		5.8	2.5	1.1	167.7			
18.0	3.0	7.6	1.8	1.1	167.1	271.0	78.0	5.9
23.0		9.1	1.5	1.2	138.0			
28.0	2.0	10.8	1.7	1.2	162.4	271.0	78.0	5.9
34.0		12.8	2.0	1.2	156.5			
38.0	3.0	13.8	1.0	1.3	117.4	271.0	77.0	5.9
43.0		15.3	1.5	1.3	140.8			
48.0	3.0	16.3	1.0	1.3	93.9	270.0	78.0	5.8
53.0		17.8	1.5	1.4	140.8			
58.0	4.0	18.8	1.0	1.4	93.9	270.0	78.0	5.8
63.0		20.3	1.5	1.4	140.8			
68.0	5.0	21.3	1.0	1.5	93.9	269.0	80.0	5.9
73.0		22.6	1.3	1.5	122.1			
78.0	7.0	23.8	1.2	1.6	112.7	270.0	80.0	6.0
83.0		24.8	1.0	1.6	93.9			
88.0	6.0	26.1	1.3	1.7	122.1	268.0	78.0	5.9
93.0	5.0	27.1	1.0	1.7	93.9			
98.0	5.0	28.1	1.0	1.8	93.9	268.0	80.0	6.0
103.0		29.1	1.0	1.8	93.9			
108.0	6.0	30.5	1.4	1.9	131.5	269.0	82.0	6.0
113.0		31.6	1.1	1.9	103.3			
118.0	6.0	32.3	0.7	2.0	65.7	269.0	77.0	5.8
123.0		33.3	1.0	2.0	93.9			
128.0	7.0	34.3	1.0	2.1	93.9	271.0	78.0	5.7
Minutes	psi	Kg	Kg	Kg Basis	Kg/sq m/hr	Deg C	psi	USGPM

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Table 2. Coal Liquids Concentration Run Number 2

Coal liquid Concentration Run Number 3; 9/30/94

Time	Delta P	Total Permeate	Delta Permeate	Conc Factor	Perm Flux	Feed Temp	Inlet Pres	Feed Flow
0.0	1.0	0.9	0.0	1.0	251.7	278.0	78.0	6.0
3.0		2.4	1.5	1.0	125.8			
8.0	3.0	3.6	1.3	1.1	176.2	277.0	80.0	6.0
13.0		5.4	1.8	1.1	125.8			
18.0	2.0	6.6	1.3	1.1	100.7	277.0	78.0	5.8
23.0		7.6	1.0	1.1	226.5			
28.0	3.0	9.9	2.2	1.2	125.8	276.0	80.0	6.1
33.0		11.1	1.3	1.2	125.8			
38.0	3.0	12.4	1.3	1.2	125.8	275.0	78.0	5.8
43.0		13.6	1.3	1.3	125.8			
48.0	3.0	14.9	1.3	1.3	125.8	275.0	79.0	5.8
53.0		16.1	1.3	1.3	75.5			
58.0	3.0	16.9	0.8	1.3	125.8	275.0	83.0	6.3
63.0		18.1	1.3	1.4	125.8			
68.0	2.0	19.4	1.3	1.4	100.7	273.0	75.0	5.6
73.0		20.4	1.0	1.4	125.8			
78.0	4.0	21.6	1.3	1.5	100.7	272.0	78.0	5.9
83.0		22.6	1.0	1.5	100.7			
88.0	4.0	23.6	1.0	1.5	100.7	272.0	78.0	5.9
93.0		24.6	1.0	1.6	100.7			
98.0	5.0	25.6	1.0	1.6	100.7	272.0	79.0	5.9
103.0		26.9	1.3	1.7	125.8			
108.0	5.0	27.9	1.0	1.7	100.7	272.0	79.0	5.8
123.0		30.4	2.5	1.8	83.9			
128.0	6.0	31.4	1.0	1.9	100.7	272.0	80.0	5.9
133.0		32.6	1.3	1.9	125.8	272.0	80.0	5.9
Minutes	psi	Kg	Kg	Kg Basis	Kg/sq m/hr	Deg C	psi	USGPM

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Table 3. Coal Liquids Concentration Run Number 3

Coal liquid Concentration Run Number 4; 10/1/94

Time	Delta P	Total Permeate	Delta Permeate	Conc Factor	Perm Flux	Feed Temp	Inlet Pres	Feed Flow
0.0	5.0	0.9	0.0	1.0	131.3	260.0	76.0	6.0
5.0		2.2	1.3	1.0	130.9			
10.0	6.0	3.4	1.2	1.1	105.1	261.0	75.0	6.0
15.0		4.4	1.0	1.1	105.1			
20.0	5.0	5.4	1.0	1.1	131.3	263.0	78.0	5.9
25.0		6.7	1.3	1.1	105.1			
30.0		7.7	1.0	1.1	131.3			
40.0	5.0	9.7	2.0	1.2	105.1	265.0	77.0	6.0
45.0		10.9	1.3	1.2	131.3			
50.0		11.9	1.0	1.2	105.1			
55.0		12.9	1.0	1.2	105.1			
60.0	5.0	13.9	1.0	1.3	105.1	267.0	79.0	6.0
72.0		16.4	2.5	1.3	109.5			
80.0	5.0	17.9	1.5	1.4	98.5	270.0	81.0	6.2
100.0	5.0	21.4	3.5	1.5	91.9	270.0	79.0	6.0
105.0		22.7	1.3	1.5	131.3			
110.0	5.0	23.4	0.8	1.5	78.8	270.0	78.0	5.8
120.0	5.0	25.4	2.0	1.6	105.1			
130.0	5.0	27.2	1.8	1.7	91.9	270.0	78.0	5.7
135.0		27.9	0.8	1.7	78.8			
140.0	5.0	28.7	0.8	1.8	78.8			
145.0	5.0	29.4	0.8	1.8	78.8	270.0	81.0	6.0
150.0		30.4	1.0	1.8	105.1			
155.0	5.0	31.4	1.0	1.9	105.1	271.0	78.0	5.8
160.0		32.4	1.0	2.0	105.1			
165.0	5.0	33.2	0.8	2.0	79.2	271.0	78.0	5.8
Minutes	psi	Kg	Kg	Kg Basis	Kg/sq m/hr	Deg C	psi	USGPM

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Table 4. Coal Liquids Concentration Run Number 4

Coal liquid Concentration Run Number 5; 10/2/94

Time	Delta P	Total Permeate	Delta Permeate	Conc Factor	Perm Flux	Feed Temp	Inlet Pres	Feed Flow
0.0	5.0	0.8	0.0	1.0	165.2	254.0	78.0	6.2
5.0		2.3	1.5	1.0	110.0			
10.0	6.0	3.3	1.0	1.1	110.0	256.0	75.0	5.9
15.0		4.3	1.0	1.1	110.0			
20.0	5.0	5.3	1.0	1.1	110.0	258.0	78.0	5.9
25.0		6.3	1.0	1.1	110.0			
30.0	6.0	7.3	1.0	1.1	110.0	260.0	79.0	6.0
35.0		8.3	1.0	1.1	110.0			
55.0	7.0	11.5	3.3	1.2	89.4	263.0	78.0	5.7
60.0		12.5	1.0	1.2	110.0			
85.0	6.0	16.8	4.3	1.3	93.5	269.0	79.0	6.0
95.0		18.5	1.8	1.4	98.2			
105.0	6.0	20.3	1.8	1.5	98.2	271.0	78.0	5.8
110.0		21.3	1.0	1.5	110.0			
120.0	7.0	22.8	1.5	1.5	82.5			
125.0	7.0	23.8	1.0	1.6	110.0	272.0	82.0	6.1
130.0		24.5	0.8	1.6	82.5			
135.0		25.3	0.8	1.6	82.5			
140.0	7.0	26.3	1.0	1.7	110.0	273.0	78.0	5.9
155.0	6.0	28.3	2.0	1.8	73.3	269.0	78.0	5.8
160.0		29.0	0.8	1.8	82.5			
170.0	7.0	30.8	1.8	1.9	98.2	269.0	78.0	5.8
175.0		31.5	0.8	1.9	82.5			
180.0	6.0	32.3	0.8	2.0	82.5	270.0	79.0	5.8
Minutes	psi	Kg	Kg	Kg Basis	Kg/sq m/hr	Deg C	psi	USGPM

37

Table 5. Coal Liquids Concentration Run Number 5
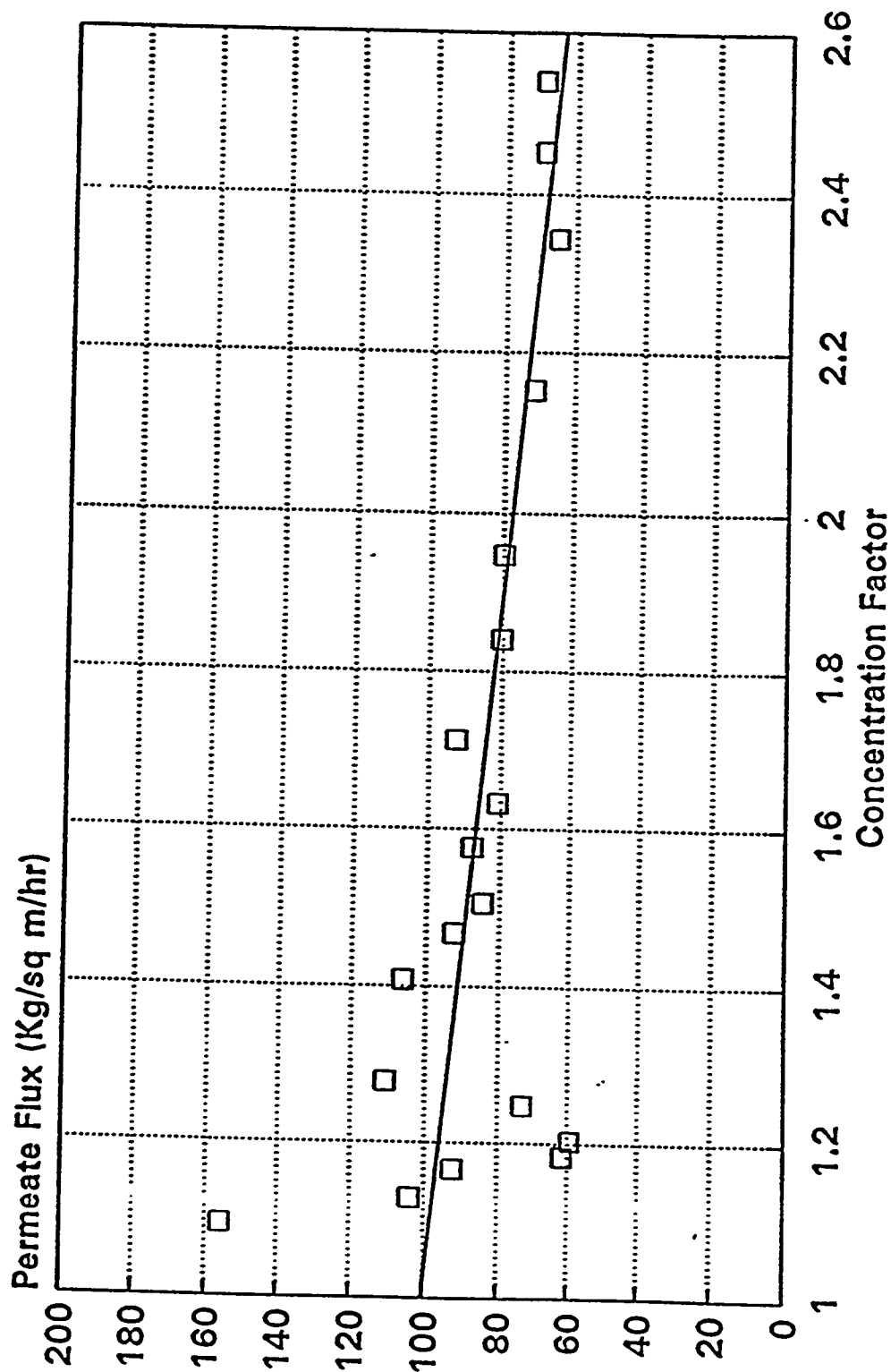
Figure 1. Process flux as a function of concentration factor for Run Number 1

Pressure Drop vs. Concentration Factor
Coal Liquids Run 1
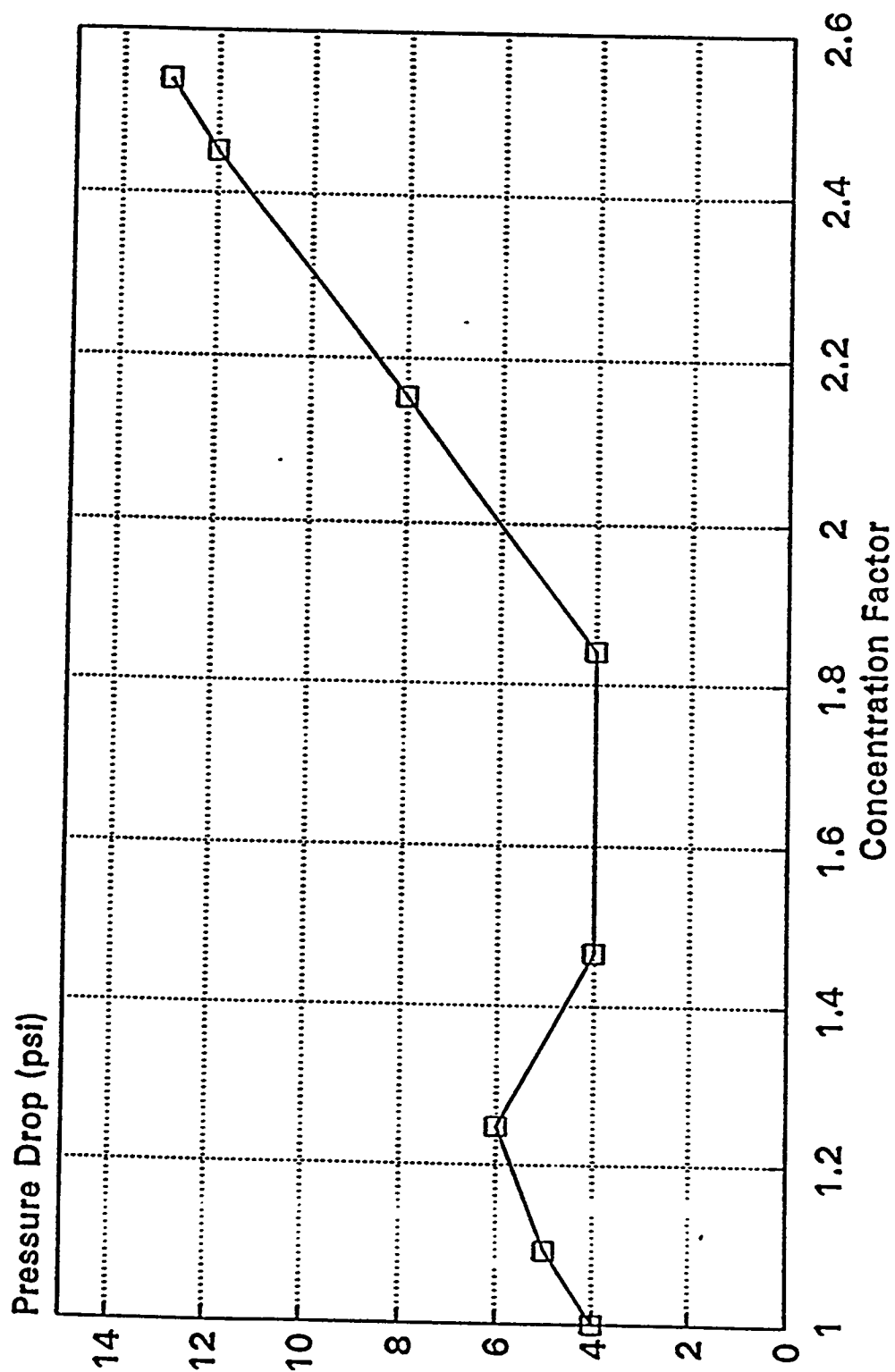
Figure 2. Module pressure drop as a function of concentration factor for Run Number 1

Flux vs. Concentration Factor
Coal Liquids Run 2

Permeate Flux (Kg/sq m/hr)

Concentration Factor

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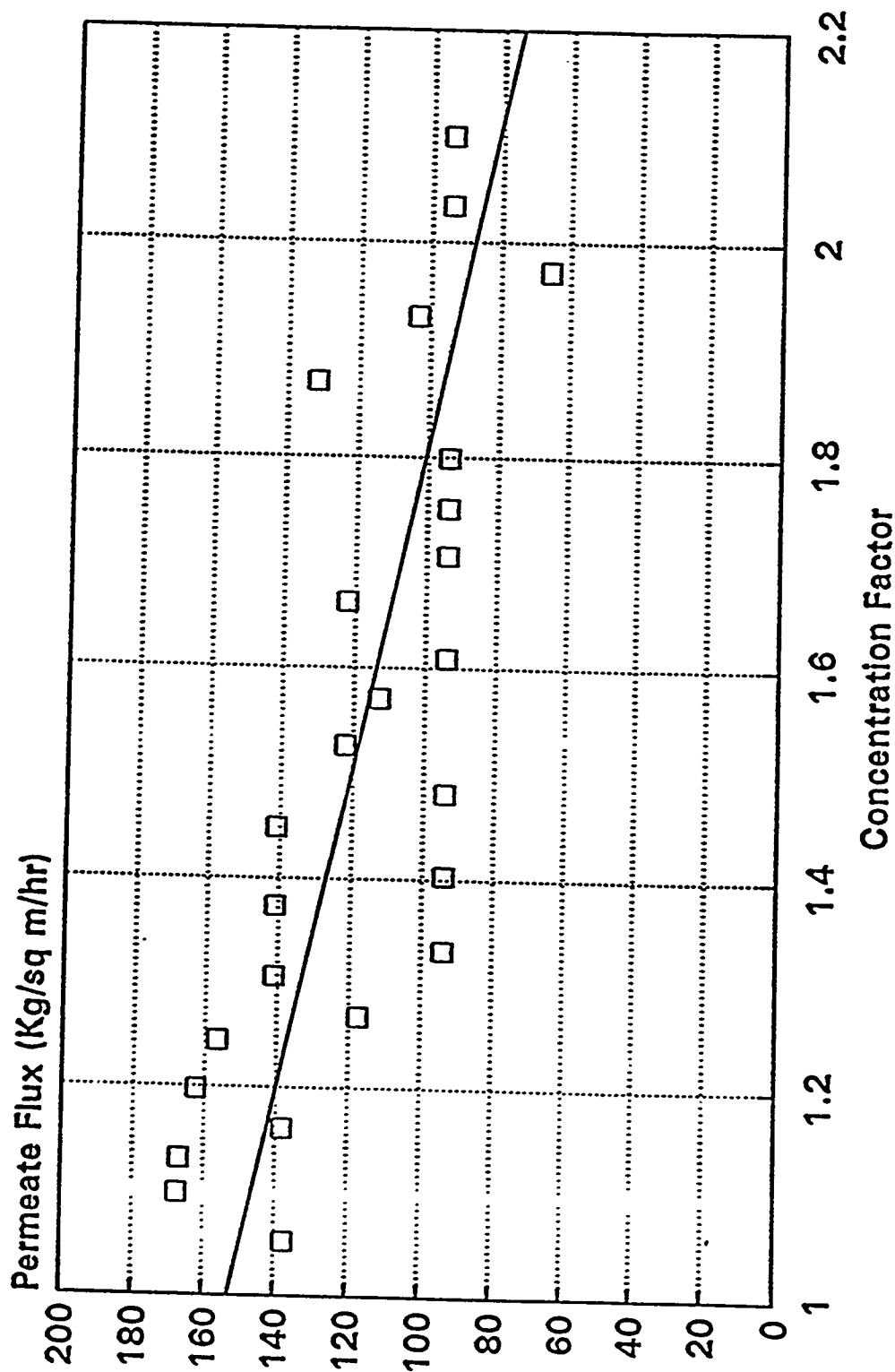
Figure 3. Process flux as a function of concentration factor for Run Number 2
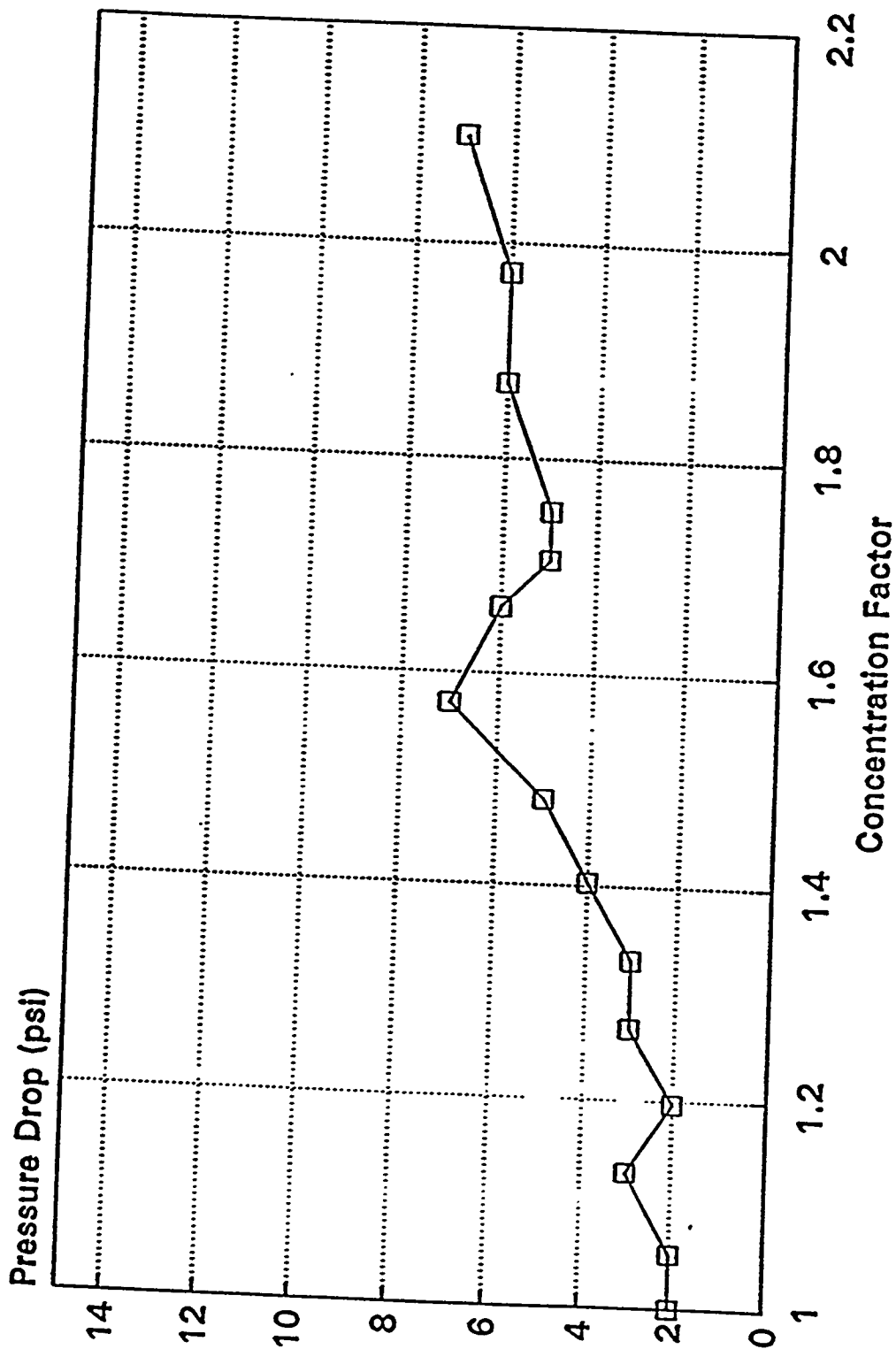
Figure 4. Module pressure drop as a function of concentration factor for Run Number 2

Flux vs. Concentration Factor
Coal Liquids Run 3
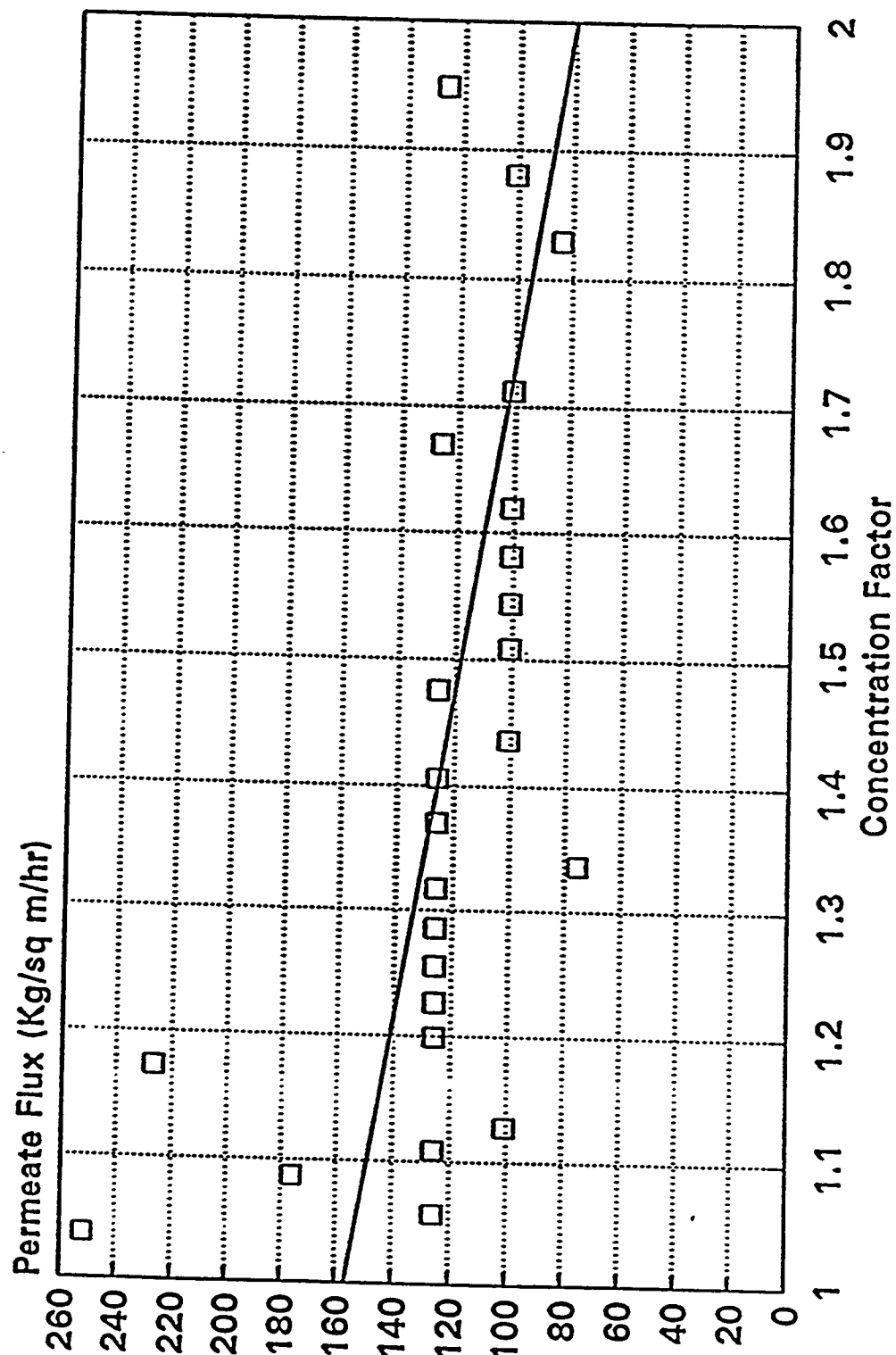
Figure 5. Process flux as a function of concentration factor for Run Number 3
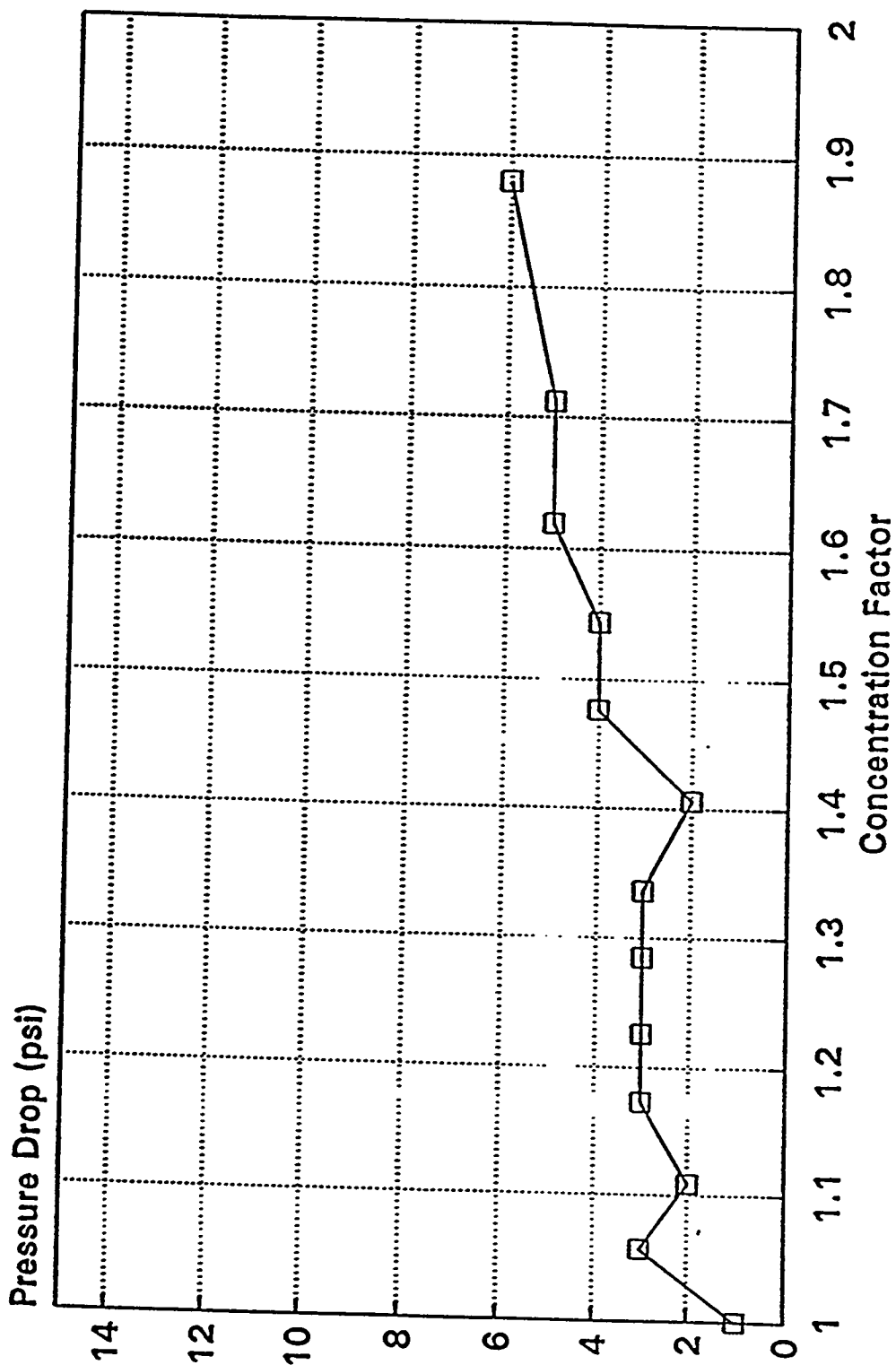
Figure 6. Module pressure drop as a function of concentration factor for Run Number 3

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Flux vs. Concentration Factor
Coal Liquids Run 4
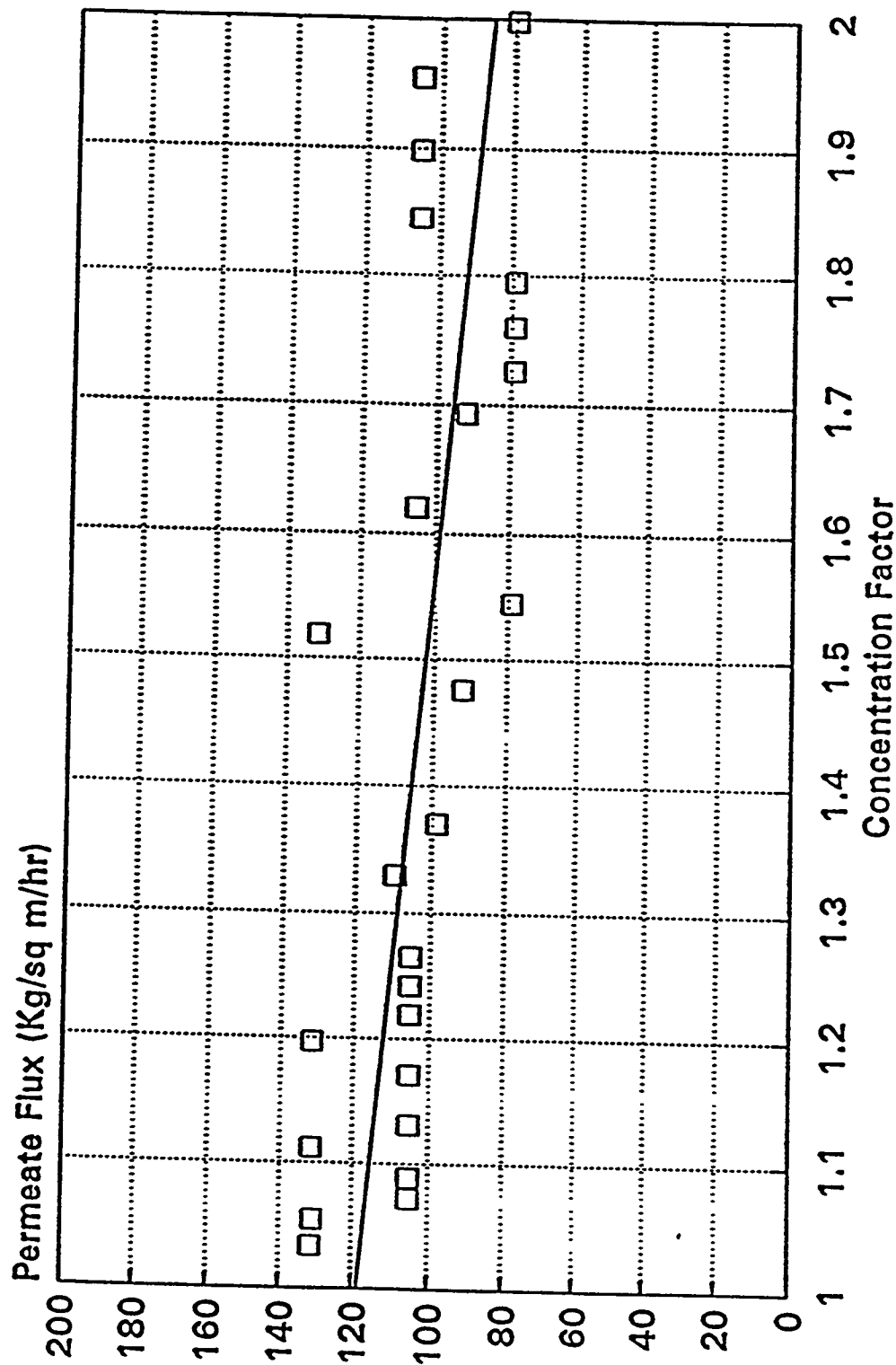
Figure 7. Process flux as a function of concentration factor for Run Number 4

Pressure Drop vs. Concentration Factor
Coal Liquids Run 4
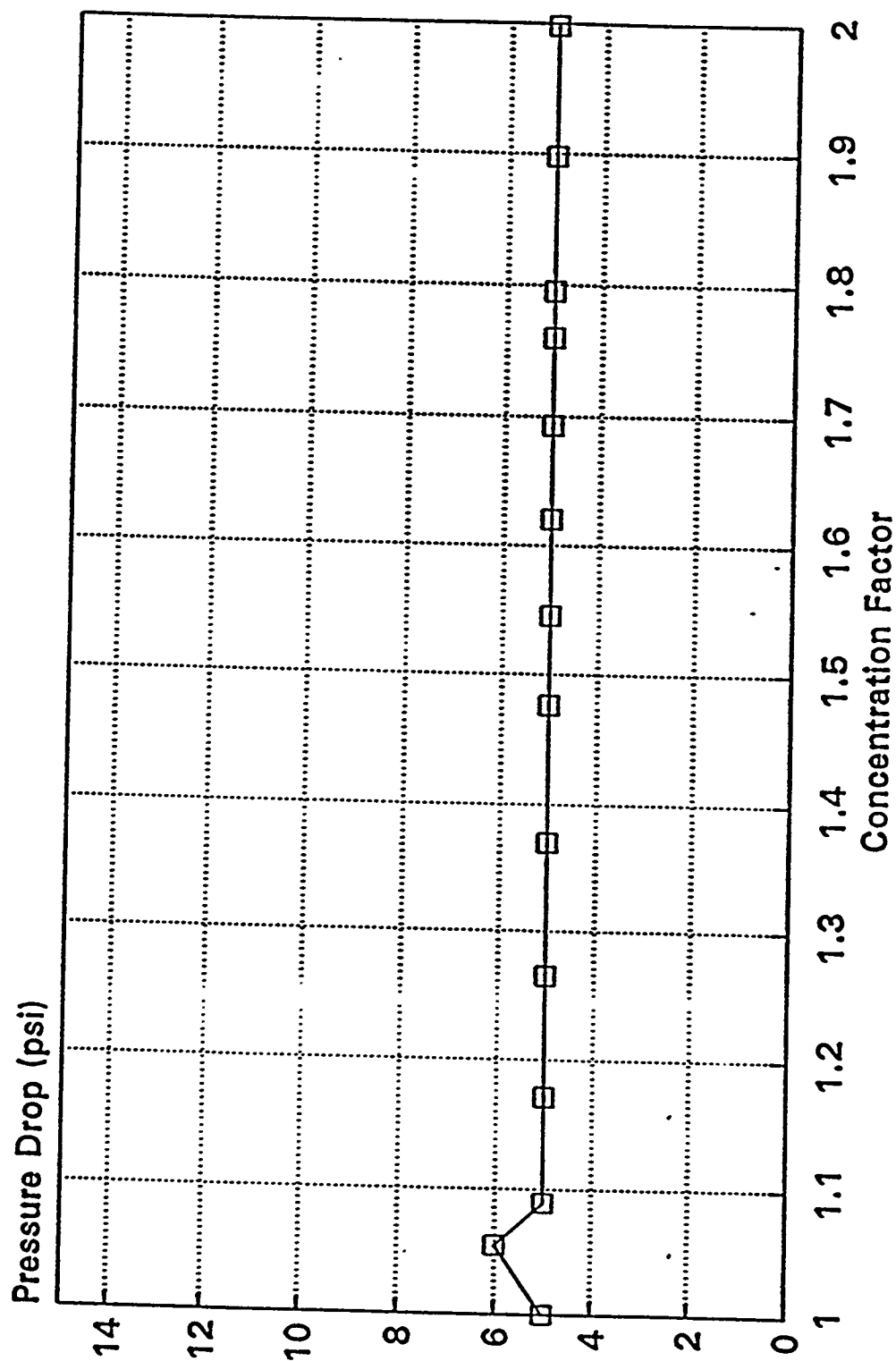
Figure 8. Module pressure drop as a function of concentration factor for Run Number 4

Flux vs. Concentration Factor
Coal Liquids Run 5
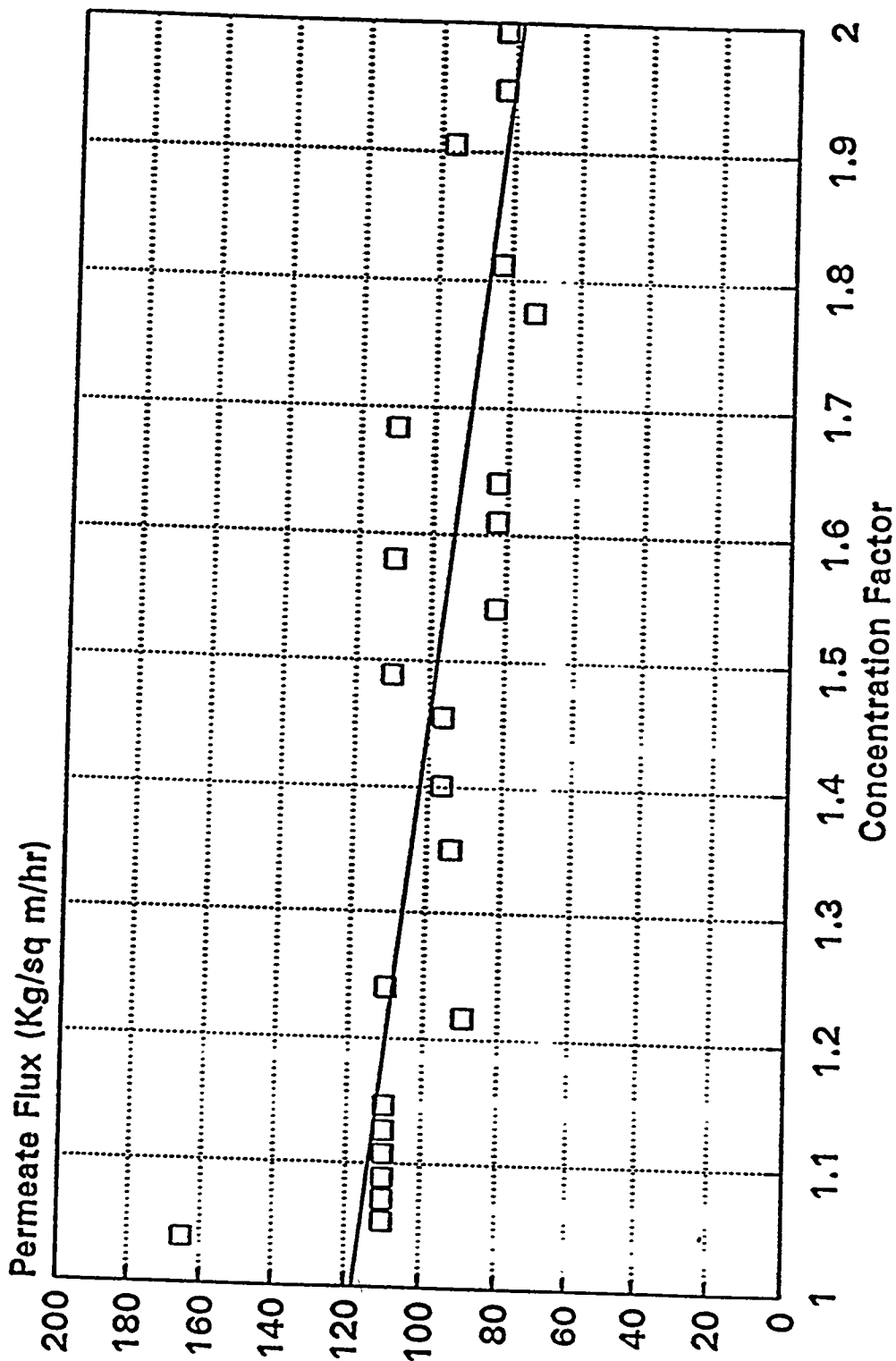
Figure 9. Process flux as a function of concentration factor for Run Number 5
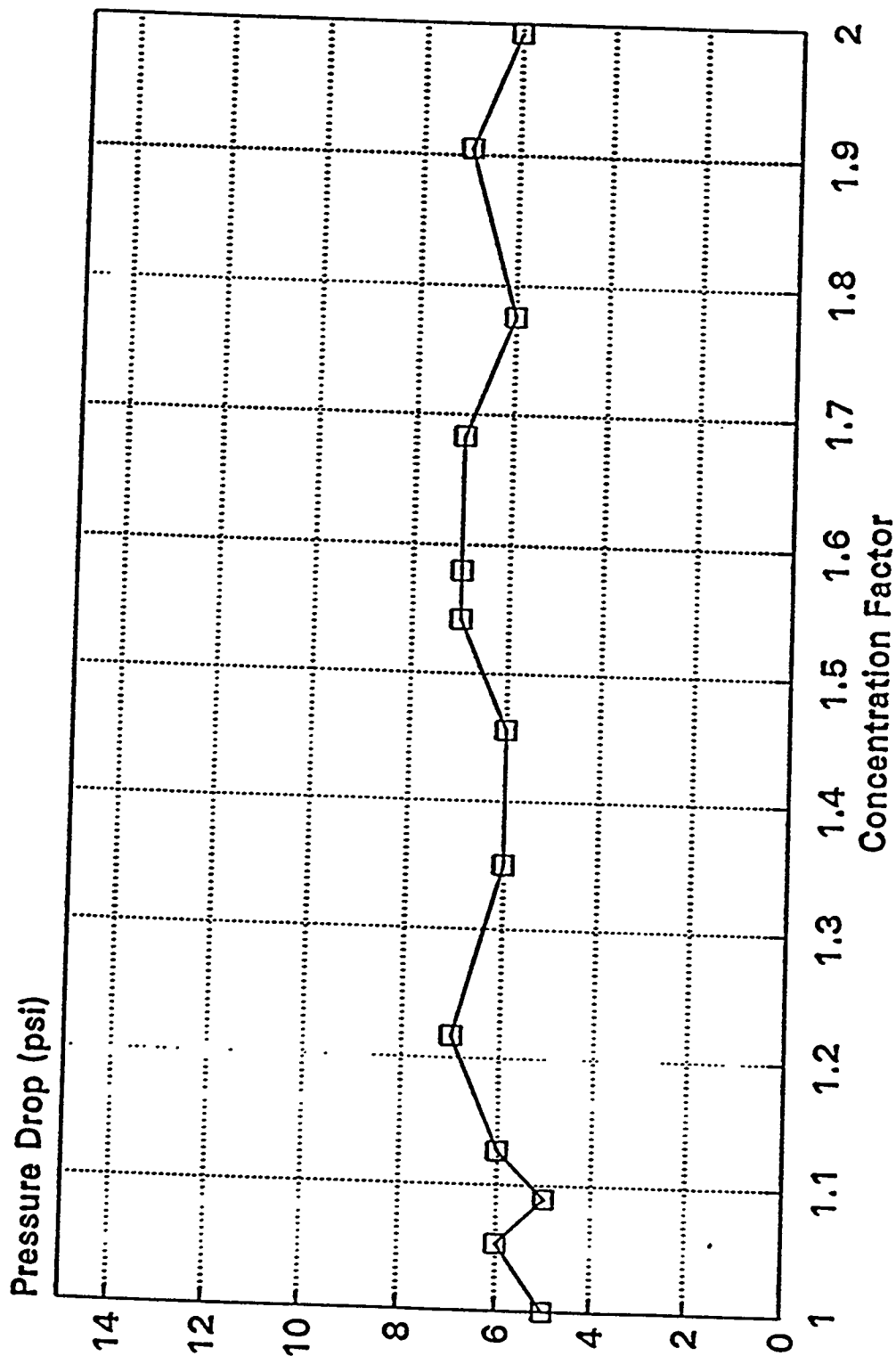
Figure 10. Module pressure drop as a function of concentration factor for Run Number 5

Attachment 2
Data Table for First Concentration Run

Attachment 2 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #1, CeraMem Module AD-2054

RUN: B86-94-03
FEED: DEASH-03 (Coal Ash liquid/VGO) (10% coal ash solids)

Ceramem Module: AD-2054
Pore Size: 0.05 micron
Area: 0.13 m2

Housing: Grapholl packing gland (70 lb. density seal rings) (45 ft.lbs. torque)

UNIT DATA

Date/Time	Time on Stream (hr)	(days)	ID	Feed Charge (kg)	Removed Sample wt (kg)	Pump Setting (Scale)	Feed Flow (USgpm)	Velocity	Temperature Element (C)	Fd Tk Skin (C)	Fd Tank Press (PSIG)	Pump Outlet Press (PSIG)	Pre-Ht Outlet Press (PSIG)	Housing Inlet (PSIG)	Housing Outlet (PSIG)	Outlet corrected (PSIG)	Module Delta Press (PSIG)	Perm Press (PSI)
(mo/dd/yr hr-m)	(hrs)	(days)		(kg)	(kg)	(Scale)	(USgpm)	n/a	(C)	(C)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSI)
9/19/94 17:15	71.1	3.0	DEASH-03	63.5		2.7	6	n/a	273	281	39	84	85	78	69	74	4	0
9/19/94 17:20	71.2	3.0	DEASH-03	63.5				6.54										
9/19/94 17:28	71.3	3.0	DEASH-03		0.5	2.7	5.8	6.33	274	281	36	82	83	78	68	73	5	0
9/19/94 17:36	71.4	3.0	DEASH-03	58.1														
9/19/94 17:44	71.6	3.0	DEASH-03	56.3														
9/19/94 17:53	71.7	3.0	DEASH-03	54.5														
9/19/94 17:59	71.8	3.0	DEASH-03	53.7														
9/19/94 18:06	71.9	3.0	DEASH-03	52.8														
9/19/94 18:16	72.1	3.0	DEASH-03	50.9		2.75	5.8	6.33	274	280	35	84	85	79	68	73	6	0
9/19/94 18:18	72.1	3.0	DEASH-03	49.7														
9/19/94 18:23	72.2	3.0	DEASH-03	45.1														
9/19/94 18:43	72.6	3.0	DEASH-03															
9/19/94 18:50	72.7	3.0	DEASH-03	43.3		2.7	6	6.54	271	278	32	82	83	78	69	74	4	0
9/19/94 18:52	72.7	3.0	DEASH-03	42.2														
9/19/94 18:58	72.8	3.0	DEASH-03	40.3														
9/19/94 19:08	73.0	3.0	DEASH-03	38.9														
9/19/94 19:16	73.1	3.0	DEASH-03	37.1														
9/19/94 19:25	73.3	3.1	DEASH-03	34.5														
9/19/94 19:40	73.5	3.1	DEASH-03															
9/19/94 19:42	73.5	3.1	DEASH-03	32.6		2.6	6	6.54	270	271	33	86	87	81	72	77	4	0
9/19/94 19:51	73.7	3.1	DEASH-03															
9/19/94 20:11	74.0	3.1	DEASH-03	29.5		2.5	5.7	6.22	272	275	32	84	85	80	67	72	8	0
9/19/94 20:28	74.3	3.1	DEASH-03	27.1														
9/19/94 20:35	74.4	3.1	DEASH-03			2.5	6	6.54	274	288	32	86	87	81	64	69	12	0
9/19/94 20:36	74.4	3.1	DEASH-03	25.9														
9/19/94 20:40	74.5	3.1	DEASH-03		0.5	2.5	6	6.54			32	87	88	81	63	68	13	0
9/19/94 20:42	74.6	3.1	DEASH-03	22.9														

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Attachment 2 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #1, CeraMem Module AD-2054

	Concentration Study Data				PERMEATE FLOW					FEED THF ANALYSIS			
Date/Time	Elapsed Time	Barrel wt.	Total Perm wt.	CF	Perm wt	Perm Time	Perm wt	Flux	Flux	Feed Sample ID	Filter paper clean	Filter paper soiled	THF Insolubles
(mo/dd/yr hr-m)	(min)	(kg)	(kg)		(g)	(min)	(g/min)	(kg/m2-d)	(kg/m2-hr)		(g)	(g)	(g/100ml)
9/19/94 17:15													
9/19/94 17:20	0	30.5	0	1									
9/19/94 17:28					161.79	0.50	323.58	3584.3	149.3	deash-28			7.1964
9/19/94 17:36	16	35.4	5.4	1.09									
9/19/94 17:44	24	37.2	7.2	1.13									
9/19/94 17:53	33	39	9	1.17									
9/19/94 17:59	39	39.8	9.8	1.18									
9/19/94 18:06	46	40.7	10.7	1.20									
9/19/94 18:16													
9/19/94 18:18	58	42.6	12.6	1.25									
9/19/94 18:23	63	43.8	13.8	1.28									
9/19/94 18:43	83	48.4	18.4	1.41									
9/19/94 18:50													
9/19/94 18:52	92	50.2	20.2	1.47									
9/19/94 18:58	98	51.3	21.3	1.50									
9/19/94 19:08	108	53.2	23.2	1.58									
9/19/94 19:16	116	54.6	24.6	1.63									
9/19/94 19:25	125	56.4	26.4	1.71									
9/19/94 19:40	140	59	29	1.84									
9/19/94 19:42													
9/19/94 19:51	151	60.9	30.9	1.95									
9/19/94 20:11	171	64	34	2.15									
9/19/94 20:28	188	66.4	36.4	2.34									
9/19/94 20:35													
9/19/94 20:36	196	67.6	37.6	2.45									
9/19/94 20:40					192.64	1.25	154.11	3692.5	153.9	deash-32	0.9138	30.2952	29.3814
9/19/94 20:42	202	68.5	38.5	2.54									

PERMEATE THF ANALYSIS

Date/Time	Permeate Sample ID	Filter paper clean (g)	Filter paper soiled (g)	THF Insolubles (g/100ml)	THF Insolubles Rejection (%)
(mo/dd/yr hr-m)					
9/19/94 17:15					
9/19/94 17:20					
9/19/94 17:28	deash-29	0.8715	0.8954	0.0239	99.67
9/19/94 17:36					
9/19/94 17:44					
9/19/94 17:53					
9/19/94 17:59					
9/19/94 18:06					
9/19/94 18:16					
9/19/94 18:18					
9/19/94 18:23					
9/19/94 18:43					
9/19/94 18:50					
9/19/94 18:52					
9/19/94 18:58					
9/19/94 19:08					
9/19/94 19:16					
9/19/94 19:25					
9/19/94 19:40					
9/19/94 19:42					
9/19/94 19:51					
9/19/94 20:11					
9/19/94 20:28					
9/19/94 20:35					
9/19/94 20:36					
9/19/94 20:40	deash-30	0.9118	0.9161	0.0043	99.99
9/19/94 20:42					

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Date/Time	Event	Comments
(mo/dd/yr hr-m)		Active membrane area used for initial flux = 0.13 m2
9/19/94 17:15	Start	
9/19/94 17:20		Took initial permeate sample for concentration study ; deash-29
9/19/94 17:28		Began concentration study - permeating to external permeation collection barrel
9/19/94 17:36		
9/19/94 17:44		
9/19/94 17:53		
9/19/94 17:59		
9/19/94 18:06		
9/19/94 18:16		
9/19/94 18:18		
9/19/94 18:23		
9/19/94 18:43		
9/19/94 18:50		
9/19/94 18:52		
9/19/94 18:58		
9/19/94 19:08		
9/19/94 19:16		
9/19/94 19:25		
9/19/94 19:40		
9/19/94 19:42		
9/19/94 19:51		
9/19/94 20:11		
9/19/94 20:28		
9/19/94 20:35		Took final permeate and feed samples for concentration study - Final Perm. sample: deash-31 for CeraMem
9/19/94 20:36		Stopped permeation - module delta pressure greater than 10 psi, Injected 43.059 kg VGO to unit - New feed wt. = 65.5 kg
9/19/94 20:40		Module inspection showed 32 plugged feed channels - active membrane area for final flux calculation = 0.061 m2
9/19/94 20:42	End	Removed module from unit - Inspection showed module failure - 2 sections of monolith cracked - module performance okay, therfore, damage occured after operation - perhaps during shut-down or dismantling.

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Attachment 3
Data Table for Second Concentration Run

Attachment 3 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #2, CeraMem Module AD-2053

RUN: B86-94-03
FEED: DEASH-03 (Coal Ash liquid/VGO) (10% coal ash solids)

CeraMem Module: AD-2053
Pore Size: 0.05 micron
Area: 0.13 m2

Housing:

Graphoil packing gland (70 lb. density seal rings) (45 ft.lbs. torque)

UNIT DATA

Date/Time	Time on Stream		ID	Feed Charge	Removed Sample wt	Pump Setting	Feed Flow	Velocity	Temperature Element	Temperature Fd Tk Skin	Fd Tank Press.	Pump Outlet Press	Pre-Ht Outlet Press	Housing Press Inlet	Housing Press Outlet	Outlet corrected	Module Delta Press	Perm Press
(mo/dd/yr hr-m)	(hrs)	(days)		(kg)	(kg)	(Scale)	(USgpm)	ft/s	(C)	(C)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSI)
Conc. Run #2																		
9/27/94 9:12	0.0	0.0	DEASH-03	65.5	1.57	2.8	5.8	6.33	271	275	35	82	83	78	71	76	2	0
9/27/94 9:18	0.1	0.0	DEASH-03	62.2		2.8	5.8	6.33	271	275	34	82	83	78	71	76	2	0
9/27/94 9:25	0.2	0.0	DEASH-03	59.7														
9/27/94 9:30	0.3	0.0	DEASH-03	57.9		2.8	5.9	6.43	271	275	32	81	82	78	70	75	3	0
9/27/94 9:35	0.4	0.0	DEASH-03	56.4														
9/27/94 9:40	0.5	0.0	DEASH-03	54.7		2.8	5.9	6.43	271	275	33	82	83	78	71	76	2	0
9/27/94 9:46	0.6	0.0	DEASH-03	52.7														
9/27/94 9:50	0.6	0.0	DEASH-03	51.7		2.8	5.9	6.43	271	274	31	81	82	77	69	74	3	0
9/27/94 9:55	0.7	0.0	DEASH-03	50.2														
9/27/94 10:00	0.8	0.0	DEASH-03	49.2		2.8	5.8	6.33	270	274	33	82	83	78	70	75	3	0
9/27/94 10:05	0.9	0.0	DEASH-03	47.7														
9/27/94 10:10	1.0	0.0	DEASH-03	46.7		2.8	5.8	6.33	270	273	33	83	84	79	70	75	4	0
9/27/94 10:15	1.1	0.0	DEASH-03	45.2														
9/27/94 10:20	1.1	0.0	DEASH-03	44.2		2.8	5.9	6.43	269	271	33	84	85	80	70	75	5	0
9/27/94 10:25	1.2	0.1	DEASH-03	42.9														
9/27/94 10:30	1.3	0.1	DEASH-03	41.7		2.7	6	6.54	270	270	32	84	85	80	68	73	7	0
9/27/94 10:35	1.4	0.1	DEASH-03	40.7														
9/27/94 10:40	1.5	0.1	DEASH-03	39.4		2.6	5.9	6.43	268	269	31	79	80	78	67	72	6	0
9/27/94 10:04	0.9	0.0	DEASH-03	38.4														
9/27/94 10:50	1.6	0.1	DEASH-03	37.4		2.6	6	6.54	268	267	31	83	84	80	70	75	5	0
9/27/94 10:55	1.7	0.1	DEASH-03	36.4														
9/27/94 11:00	1.8	0.1	DEASH-03	35.2		2.6	6	6.54	269	266	30	83	84	82	71	76	6	0
9/27/94 11:05	1.9	0.1	DEASH-03	33.9														
9/27/94 11:10	2.0	0.1	DEASH-03	33.2		2.6	5.8	6.33	269	266	32	78	79	77	66	71	6	0
9/27/94 11:05	1.9	0.1	DEASH-03	32.2														
9/27/94 11:20	2.1	0.1	DEASH-03	31.2	0.392	2.6	5.7	6.22	271	268	31	79	80	78	66	71	7	0

Attachment 3 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #2, CeraMem Module AD-2053

	Concentration Study Data				PERMEATE FLOW					FEED THF ANALYSIS			
Date/Time	Elapsed Time	Barrel wt.	Total Perm wt.	CF	Perm wt	Perm Time	Perm wt	Flux	Flux	Feed Sample ID	Filter paper clean	Filter paper soiled	THF Insolubles
(mo/dd/yr hr:m)	(min)	(kg)	(kg)		(g)	(min)	(g/min)	(kg/m2-d)	(kg/m2-hr)		(g)	(g)	(g/100ml)
Conc. Run #2													
9/27/94 9:12	0	30.25	1.57	1.02	345.76	0.67	518.64	5745.0	239.4	deash-33	0.8819	10.3215	9.4396
9/27/94 9:18	6	32	3.32	1.05									
9/27/94 9:25	13	34.5	5.82	1.10									
9/27/94 9:30	18	36.25	7.57	1.13									
9/27/94 9:35	23	37.75	9.07	1.16									
9/27/94 9:40	28	39.5	10.82	1.20									
9/27/94 9:46	34	41.5	12.82	1.24									
9/27/94 9:50	38	42.5	13.82	1.27									
9/27/94 9:55	43	44	15.32	1.31									
9/27/94 10:00	48	45	16.32	1.33									
9/27/94 10:05	53	46.5	17.82	1.37									
9/27/94 10:10	58	47.5	18.82	1.40									
9/27/94 10:15	63	49	20.32	1.45									
9/27/94 10:20	68	50	21.32	1.48									
9/27/94 10:25	73	51.25	22.57	1.53									
9/27/94 10:30	78	52.5	23.82	1.57									
9/27/94 10:35	83	53.5	24.82	1.61									
9/27/94 10:40	88	54.75	26.07	1.66									
9/27/94 10:04	93	55.75	27.07	1.70									
9/27/94 10:50	98	56.75	28.07	1.75									
9/27/94 10:55	103	57.75	29.07	1.80									
9/27/94 11:00	108	59	30.32	1.86									
9/27/94 11:05	113	60.25	31.57	1.93									
9/27/94 11:10	118	61	32.32	1.97									
9/27/94 11:05	123	62	33.32	2.04									
9/27/94 11:20	128	63	34.32	2.10	401.24	1.75	229.28	2769.7	115.4	deash-36	0.8897	23.02	22.1303
	FINAL	63.25	34.962	2.14									

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Attachment 3 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #2, CeraMem Module AD-2053

PERMEATE THF ANALYSIS

Date/Time (mo/dd/yr hr:m)	Permeate Sample ID	Filter paper clean (g)	Filter paper soiled (g)	THF Insolubles (g/100ml)	THF Insolubles Rejection (%)	Event	Comments
Conc. Run #2						Run #2	
9/27/94 9:12	deash-34					Start	Took initial permeate flux sample (deash-34) then began permeating to permeate collection barrel.
9/27/94 9:18							
9/27/94 9:25							
9/27/94 9:30							
9/27/94 9:35							
9/27/94 9:40							
9/27/94 9:46							
9/27/94 9:50							
9/27/94 9:55							
9/27/94 10:00							
9/27/94 10:05							
9/27/94 10:10							
9/27/94 10:15							
9/27/94 10:20							
9/27/94 10:25							
9/27/94 10:30							
9/27/94 10:35							
9/27/94 10:40							
9/27/94 10:04							
9/27/94 10:50							
9/27/94 10:55							
9/27/94 11:00							
9/27/94 11:05							
9/27/94 11:10							
9/27/94 11:05							
9/27/94 11:20	deash-35	0.9143	0.9161	0.0018	99.99	END	Took final permeate flux sample (deash-35) then ceased concentration study. Injected 36.112 kg VGO to unit - New feed wt. = 67 kg. Module inspection showed 5 plugged feed channels - membrane area for final flux calculation = 0.1193 m2

Attachment 4
Data Table for Third Concentration Run

Attachment 4 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #3, CeraMem Module AD-2053

RUN: B86-94-03
FEED: DEASH-03 (Coal Ash liquid/VGO) (10% coal ash solids)
Housing: CeraMem Module AD-2053 Pore Size: 0.05 micron Area: 0.13 m2
Graphoil packing gland (70 lb. density seal rings) (45 ft.lbs. torque)

UNIT DATA

Date/Time	Time on Stream		ID	Feed Charge	Removed Sample wt	Pump Setting	Feed Flow	Velocity	Temperature Element	Temperature Fd Tk Skin	Fd Tank Press.	Pump Outlet Press	Pre-Ht Outlet Press	Housing Press Inlet	Housing Press Outlet	Outlet corrected	Module Delta Press	Perm Press
mo/dd/yr hr-m.	(hrs)	(days)		(kg)	(kg)	(Scale)	(USgpm)	ft/s	(C)	(C)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSIG)	(PSI)
Conc. Run #3																		
9/30/94 7:27	0.0	0.0	DEASH-03	67.1	0.86	2.8	6	6.54	278	284	36	79	80	76	70	75	1	0
9/30/94 7:30	0.0	0.0	DEASH-03	64.7														
9/30/94 7:35	0.1	0.0	DEASH-03	63.4		2.75	6	6.54	277	283	35	84	86	80	72	77	3	0
9/30/94 7:40	0.2	0.0	DEASH-03	61.7														
9/30/94 7:45	0.3	0.0	DEASH-03	60.4		2.75	5.8	6.33	277	282	34	83	84	78	71	76	2	0
9/30/94 7:50	0.4	0.0	DEASH-03	59.7														
9/30/94 7:55	0.5	0.0	DEASH-03	57.2		2.7	6.1	6.65	276	281	34	81	83	80	72	77	3	0
9/30/94 8:00	0.6	0.0	DEASH-03	55.9														
9/30/94 8:05	0.6	0.0	DEASH-03	54.7		2.7	5.8	6.33	275	280	34	81	83	78	70	75	3	0
9/30/94 8:10	0.7	0.0	DEASH-03	53.4														
9/30/94 8:15	0.8	0.0	DEASH-03	52.2		2.7	5.8	6.33	275	279	33	81	83	79	71	76	3	0
9/30/94 8:20	0.9	0.0	DEASH-03	50.9														
9/30/94 8:25	1.0	0.0	DEASH-03	50.2		2.6	6.3	6.87	275	278	33	86	88	83	75	80	3	0
9/30/94 8:30	1.0	0.0	DEASH-03	48.9														
9/30/94 8:35	1.1	0.0	DEASH-03	47.7		2.6	5.6	6.11	273	276	33	77	79	75	68	73	2	0
9/30/94 8:40	1.2	0.1	DEASH-03	46.7														
9/30/94 8:45	1.3	0.1	DEASH-03	45.4		2.7	5.9	6.43	272	275	33	80	82	78	69	74	4	0
9/30/94 8:50	1.4	0.1	DEASH-03	44.4														
9/30/94 8:55	1.5	0.1	DEASH-03	43.4		2.7	6	6.54	272	273	34	81	83	79	70	75	4	0
9/30/94 9:00	1.5	0.1	DEASH-03	42.4														
9/30/94 9:05	1.6	0.1	DEASH-03	41.4		2.7	5.9	6.43	272	271	33	80	82	79	69	74	5	0
9/30/94 9:10	1.7	0.1	DEASH-03	40.2														
9/30/94 9:15	1.8	0.1	DEASH-03	39.2		2.7	5.8	6.33	272	270	33	79	81	79	69	74	5	0
9/30/94 9:30	2.1	0.1	DEASH-03	36.7														
9/30/94 9:35	2.1	0.1	DEASH-03	35.7		2.7	5.9	6.43	272	269	34	82	84	80	69	74	6	0
9/30/94 9:40	2.2	0.1	DEASH-03	34.4	0.92	2.7	5.9	6.43	272	269	34	82	84	80	69	74	6	0

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Attachment 4 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #3, CeraMem Module AD-2053

	Concentration Study Data				PERMEATE FLOW					FEED THF ANALYSIS			
Date/Time	Elapsed Time	Barrel wt.	Total Perm wt.	CF	Perm wt	Perm Time	Perm wt	Flux	Flux	Feed Sample ID	Filter paper clean	Filter paper soiled	THF Insolubles
(mo/dd/yr hr-m)	(min)	(kg)	(kg)		(g)	(min)	(g/min)	(kg/m2-d)	(kg/m2.hr)		(g)	(g)	(g/100ml)
Conc. Run #3													
9/30/94 7:27	0	30.5	0.86	1.01	321.06	0.83	385.27	4654.0	193.9	deash-38	0.8793	9.5507	8.6714
9/30/94 7:30	3	32	2.36	1.04									
9/30/94 7:35	8	33.25	3.61	1.06									
9/30/94 7:40	13	35	5.36	1.09									
9/30/94 7:45	18	36.25	6.61	1.11									
9/30/94 7:50	23	37	7.36	1.12									
9/30/94 7:55	28	39.5	9.86	1.17									
9/30/94 8:00	33	40.75	11.11	1.20									
9/30/94 8:05	38	42	12.36	1.23									
9/30/94 8:10	43	43.25	13.61	1.25									
9/30/94 8:15	48	44.5	14.86	1.28									
9/30/94 8:20	53	45.75	16.11	1.32									
9/30/94 8:25	58	46.5	16.86	1.34									
9/30/94 8:30	63	47.75	18.11	1.37									
9/30/94 8:35	68	49	19.36	1.41									
9/30/94 8:40	73	50	20.36	1.44									
9/30/94 8:45	78	51.25	21.61	1.48									
9/30/94 8:50	83	52.25	22.61	1.51									
9/30/94 8:55	88	53.25	23.61	1.54									
9/30/94 9:00	93	54.25	24.61	1.58									
9/30/94 9:05	98	55.25	25.61	1.62									
9/30/94 9:10	10	56.5	26.86	1.67									
9/30/94 9:15	108	57.5	27.86	1.71									
9/30/94 9:30	123	60	30.36	1.83									
9/30/94 9:35	128	61	31.36	1.88									
9/30/94 9:40	133	62.25	32.61	1.95	397.75	2.00	198.88	2507.8	104.5	Deash-40	0.9158	18.0028	17.087
	FINAL	62.5	33.78	2.02									

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PERMEATE THF ANALYSIS

Date/Time (mo/dd/yr hr-m) Conc. Run #3	Permeate Sample ID	Filter paper clean (g)	Filter paper soiled (g)	THF Insolubles (g/100ml)	THF Insolubles Rejection (%)	Event	Comments
9/30/94 7:27	deash-37	0.8951	0.9104	0.0153	99.82	Start	Took initial permeate flux sample (deash-37) then began permeating to permeate collection barrel. Membrane area for initial flux calculation = 0.1193 m2
9/30/94 7:30							
9/30/94 7:35							
9/30/94 7:40							
9/30/94 7:45							
9/30/94 7:50							
9/30/94 7:55							
9/30/94 8:00							
9/30/94 8:05							
9/30/94 8:10							
9/30/94 8:15							
9/30/94 8:20							
9/30/94 8:25							
9/30/94 8:30							
9/30/94 8:35							
9/30/94 8:40							
9/30/94 8:45							
9/30/94 8:50							
9/30/94 8:55							
9/30/94 9:00							
9/30/94 9:05							
9/30/94 9:10							
9/30/94 9:15							
9/30/94 9:30							
9/30/94 9:35							
9/30/94 9:40	Deash-39	0.9276	0.9328	0.0052	99.97	END	Took final permeate flux sample (deash-39) then ceased concentration study. Injected 34.04 kg VGO to unit - New feed wt. = 66.45 kg Estimate 7.3 plugged feed channels - membrane area for final flux calculation = 0.1142 m2

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Attachment 5
Data Table for Fourth Concentration Run

Attachment 5 - CeraMem Deashing Program - SRC B88 Run B88-94-03, Concentration Run #4, CeraMem Module AD-2053

RUN: B86-94-03
FEED: DEASH-03 (Coal Ash liquid/VGO) (10% coal ash solids)

CeraMem Module: AD-2053
Pore Size: 0.05 micron
Area: 0.13 m2

Housing:

Grapholl packing gland (70 lb. density seal rings) (45 ft.lbs. torque)

UNIT DATA

Date/Time (mo/dd/yr hr-m)	Time on Stream (hrs)	(days)	ID	Feed Charge (kg)	Removed Sample wt (kg)	Pump Setting (Scale)	Feed Flow (USgpm)	Velocity (ft/s)	Temp Element (C)	Temp Fd Tk Skin (C)	Fd Tank Press (PSIG)	Pump Outlet Press (PSIG)	Pre-Ht Outlet Press (PSIG)	Housing Press Inlet (PSIG)	Housing Press Outlet (PSIG)	Outlet corrected (PSIG)	Module Delta Press (PSIG)	Perm Press (PSI)
Conc. Run #4																		
10/1/94 8:00	0.0	0.0	DEASH-03	66.5	0.94	2.8	6	6.54	260	256	37	78	80	76	66	71	5	0
10/1/94 8:05	0.1	0.0	DEASH-03	64.3														
10/1/94 8:10	0.2	0.0	DEASH-03	63.0		2.8	6	6.54	261	258	36	77	79	75	64	69	6	0
10/1/94 8:15	0.2	0.0	DEASH-03	62.0														
10/1/94 8:20	0.3	0.0	DEASH-03	61.0		2.8	5.9	6.43	263	260	38	80	82	78	68	73	5	0
10/1/94 8:25	0.4	0.0	DEASH-03	59.8														
10/1/94 8:30	0.5	0.0	DEASH-03	58.8														
10/1/94 8:40	0.7	0.0	DEASH-03	56.8		2.7	6	6.54	265	262	36			77	67	72	5	0
10/1/94 8:45	0.8	0.0	DEASH-03	55.5														
10/1/94 8:50	0.8	0.0	DEASH-03	54.5														
10/1/94 8:55	0.9	0.0	DEASH-03	53.5														
10/1/94 9:00	1.0	0.0	DEASH-03	52.5		2.8	6	6.54	267	264	38			79	69	74	5	0
10/1/94 9:12	1.2	0.0	DEASH-03	50.0														
10/1/94 9:20	1.3	0.1	DEASH-03	48.5		2.8	6.2	6.76	270	267	36			81	71	76	5	0
10/1/94 9:40	1.7	0.1	DEASH-03	45.0		2.75	6	6.54	270	268.	35			79	69	74	5	0
10/1/94 9:45	1.7	0.1	DEASH-03	43.8														
10/1/94 9:50	1.8	0.1	DEASH-03	43.0		2.8	5.8	6.33	270	268	36			78	68	73	5	0
10/1/94 10:00	2.0	0.1	DEASH-03	41.0														
10/1/94 10:10	2.2	0.1	DEASH-03	39.3		2.8	5.7	6.22	270	268	39			78	68	73	5	0
10/1/94 10:15	2.3	0.1	DEASH-03	38.5														
10/1/94 10:20	2.3	0.1	DEASH-03	37.8														
10/1/94 10:25	2.4	0.1	DEASH-03	37.0		2.8	6	6.54	270	267	38			81	71	76	5	0
10/1/94 10:30	2.5	0.1	DEASH-03	36.0														
10/1/94 10:35	2.6	0.1	DEASH-03	35.0		2.7	5.8	6.33	271	268	38			76	66	71	5	0
10/1/94 10:40	2.7	0.1	DEASH-03	34.0														
10/1/94 10:45	2.7	0.1	DEASH-03	33.3	0.76	2.8	5.8	6.33	271	270	38			78	68	73	5	0

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Attachment 6 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #4, CeraMem Module AD-2053

	Concentration Study Data				PERMEATE FLOW					FEED THF ANALYSIS			
Date/Time	Elapsed Time	Barrel wt.	Total Perm wt.	CF	Perm wt	Perm Time	Perm wt	Flux	Flux	Feed Sample ID	Filter paper clean	Filter paper soiled	THF Insolubles
(mo/dd/yr hr-m)	(min)	(kg)	(kg)		(g)	(min)	(g/min)	(kg/m2-d)	(kg/m2-hr)		(g)	(g)	(g/100ml)
Conc. Run #4													
10/1/94 8:00	0	30.5	0.94	1.01	329.58	0.92	359.54	4533.8	188.9	deash-41	0.9161	8.7075	7.7914
10/1/94 8:05	5	31.75	2.19	1.03									
10/1/94 8:10	10	33	3.44	1.05									
10/1/94 8:15	15	34	4.44	1.07									
10/1/94 8:20	20	35	5.44	1.09									
10/1/94 8:25	25	36.25	6.69	1.11									
10/1/94 8:30	30	37.25	7.69	1.13									
10/1/94 8:40	40	39.25	9.69	1.17									
10/1/94 8:45	45	40.5	10.94	1.20									
10/1/94 8:50	50	41.5	11.94	1.22									
10/1/94 8:55	55	42.5	12.94	1.24									
10/1/94 9:00	60	43.5	13.94	1.27									
10/1/94 9:12	72	46	16.44	1.33									
10/1/94 9:20	80	47.5	17.94	1.37									
10/1/94 9:40	100	51	21.44	1.48									
10/1/94 9:45	105	52.25	22.69	1.52									
10/1/94 9:50	110	53	23.44	1.54									
10/1/94 10:00	120	55	25.44	1.62									
10/1/94 10:10	130	56.75	27.19	1.69									
10/1/94 10:15	135	57.5	27.94	1.73									
10/1/94 10:20	140	58.25	28.69	1.76									
10/1/94 10:25	145	59	29.44	1.80									
10/1/94 10:30	150	60	30.44	1.85									
10/1/94 10:35	155	61	31.44	1.90									
10/1/94 10:40	160	62	32.44	1.95									
10/1/94 10:45	165	62.75	33.19	2.00	372.19	2.00	186.10	2454.6	102.3	deash-44	0.9206	17.4566	16.536
FINAL		63	34.20	2.06									

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Attachment 5 - CeraMem Deashing Program - SRC B88 Run B88-94-03, Concentration Run #4, CeraMem Module AD-2053

PERMEATE THF ANALYSIS

Date/Time (mo/dd/yr hr-m)	Permeate Sample ID	Filter paper clean (g)	Filter paper soiled (g)	THF Insolubles (g/100ml)	THF Insolubles Rejection (%)	Event	Comments
Conc. Run #4							
10/1/94 8:00	deash-42	0.8894	0.9060	0.0166	99.79	Start	Took initial permeate flux sample (deash-42) then began permeating to permeate collection barrel. Membrane area for initial flux calculation = 0.1142 m2
10/1/94 8:05							
10/1/94 8:10							
10/1/94 8:15							
10/1/94 8:20							
10/1/94 8:25							
10/1/94 8:30							
10/1/94 8:40							
10/1/94 8:45							
10/1/94 8:50							
10/1/94 8:55							
10/1/94 9:00							
10/1/94 9:12							
10/1/94 9:20							
10/1/94 9:40							
10/1/94 9:45							
10/1/94 9:50							
10/1/94 10:00							
10/1/94 10:10							
10/1/94 10:15							
10/1/94 10:20							
10/1/94 10:25							
10/1/94 10:30							
10/1/94 10:35							
10/1/94 10:40							
10/1/94 10:45	deash-43	0.8852	0.8910	0.0058	99.96	End	Took final permeate flux sample (deash-43) then ceased concentration study. Injected 32.943 kg VGO to unit - New feed wt. = 64.8 kg. Estimate 9.6 plugged feed channels - membrane area for final flux calculation = 0.1092 m2

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Attachment 6
Data Table for Fifth Concentration Run

Attachment 6 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #5, CeraMem Module AD-2053

RUN: B86-94-03
FEED: DEASH-03 (Coal Ash liquid/VGO) (10% coal ash solids)

Ceramem Module: AD-2053
Pore Size: 0.05 micron
Area: 0.13 m2

Housing:

Graphoil packing gland (70 lb. density seal rings) (45 ft.lbs. torque)

UNIT DATA

Date/Time (mo/dd/yr hr-m)	Time on Stream (hrs)	Time on Stream (days)	ID	Feed Charge (kg)	Removed Sample wt (kg)	Pump Setting (Scale)	Feed Flow (USgpm)	Velocity (f/s)	Temperature Element (C)	Temperature Fd Tk Skin (C)	Fd Tank Press. (PSIG)	Pump Outlet Press (PSIG)	Pre-Ht Outlet Press (PSIG)	Housing Press Inlet (PSIG)	Housing Press Outlet (PSIG)	Outlet corrected (PSIG)	Module Delta Press (PSIG)	Perm Press (PSI)
Conc. Run #5																		
10/2/94 7:50	0.0	0.0	DEASH-03	64.8	0.79	3.3	6.2	6.76	254	250	42	80	82	78	68	73	5	0
10/2/94 7:55	0.1	0.0	DEASH-03	62.5														0
10/2/94 8:00	0.2	0.0	DEASH-03	61.5		3	5.9	6.43	256	252	41			75	64	69	6	0
10/2/94 8:05	0.2	0.0	DEASH-03	60.5														0
10/2/94 8:10	0.3	0.0	DEASH-03	59.5		3	5.9	6.43	258	255	42			76	66	71	5	0
10/2/94 8:15	0.4	0.0	DEASH-03	58.5														0
10/2/94 8:20	0.5	0.0	DEASH-03	57.5		3	6	6.54	260	257	42			79	68	73	6	0
10/2/94 8:25	0.6	0.0	DEASH-03	56.5														
10/2/94 8:45	0.9	0.0	DEASH-03	53.3		3	5.7	6.22	263	260	42			76	64	69	7	0
10/2/94 8:50	1.0	0.0	DEASH-03	52.3														
10/2/94 9:15	1.4	0.1	DEASH-03	48.0		2.75	6	6.54	268	265	42			79	68	73	6	0
10/2/94 9:25	1.6	0.1	DEASH-03	46.3														
10/2/94 9:35	1.7	0.1	DEASH-03	44.5		2.75	5.8	6.33	271	267	40			76	65	70	6	0
10/2/94 9:40	1.8	0.1	DEASH-03	43.5														
10/2/94 9:50	2.0	0.1	DEASH-03	42.0														
10/2/94 9:55	2.1	0.1	DEASH-03	41.0		2.7	6.1	6.65	272	269	44			82	70	75	7	0
10/2/94 10:00	2.2	0.1	DEASH-03	40.3														
10/2/94 10:05	2.3	0.1	DEASH-03	39.5														
10/2/94 10:10	2.3	0.1	DEASH-03	38.5		2.8	5.9	6.43	273	269	42			78	66	71	7	0
10/2/94 10:25	2.6	0.1	DEASH-03	36.5		2.8	5.8	6.33	269	265	43			78	67	72	6	0
10/2/94 10:30	2.7	0.1	DEASH-03	35.8														
10/2/94 10:40	2.8	0.1	DEASH-03	34.0		2.8	5.8	6.33	269	269	43			78	66	71	7	0
10/2/94 10:45	2.9	0.1	DEASH-03	33.3														
10/2/94 10:50	3.0	0.1	DEASH-03	32.5	0.75	2.8	5.8	6.33	270	271	44			79	68	73	6	0

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Attachment 8 - CeraMem Deashing Program - SRC B86 Run B86-94-03, Concentration Run #5, CeraMem Module AD-2053

	Concentration Study Data				PERMEATE FLOW					FEED THF ANALYSIS			
Date/Time	Elapsed Time	Barrel wt.	Total Perm wt.	CF	Perm wt	Perm Time	Perm wt	Flux	Flux	Feed Sample ID	Filter paper clean	Filter paper soiled	THF Insolubles
(mo/dd/yr hr-m)	(min)	(kg)	(kg)		(g)	(min)	(g/min)	(kg/m2-d)	(kg/m2.hr)		(g)	(g)	(g/100ml)
Conc. Run #5													
10/2/94 7:50	0	30.5	0.79	1.01	325.91	1.00	325.91	4298.8	179.1	deash-45	0.8798	8.6969	7.8171
10/2/94 7:55	5	32	2.29	1.04									
10/2/94 8:00	10	33	3.29	1.05									
10/2/94 8:05	15	34	4.29	1.07									
10/2/94 8:10	20	35	5.29	1.09									
10/2/94 8:15	25	36	6.29	1.11									
10/2/94 8:20	30	37	7.29	1.13									
10/2/94 8:25	35	38	8.29	1.15									
10/2/94 8:45	55	41.25	11.54	1.22									
10/2/94 8:50	60	42.25	12.54	1.24									
10/2/94 9:15	85	46.5	16.79	1.35									
10/2/94 9:25	95	48.25	18.54	1.40									
10/2/94 9:35	105	50	20.29	1.46									
10/2/94 9:40	110	51	21.29	1.49									
10/2/94 9:50	120	52.5	22.79	1.54									
10/2/94 9:55	125	53.5	23.79	1.58									
10/2/94 10:00	130	54.25	24.54	1.61									
10/2/94 10:05	135	55	25.29	1.64									
10/2/94 10:10	140	56	26.29	1.68									
10/2/94 10:25	155	58	28.29	1.77									
10/2/94 10:30	160	58.75	29.04	1.81									
10/2/94 10:40	170	60.5	30.79	1.91									
10/2/94 10:45	175	61.25	31.54	1.95									
10/2/94 10:50	180	62	32.29	1.99	387.45	2.50	154.98	2146.5	89.4	deash-48	0.9046	19.7014	18.7968
	FINAL	62.25	33.29	2.06									

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Attachment 6 - CeraMem Deashing Program - SRC B88 Run B86-94-03, Concentration Run #5, CeraMem Module AD-2053

PERMEATE THF ANALYSIS

Date/Time (mo/dd/yr hr-m)	Permeate Sample ID	Filter paper clean (g)	Filter paper soiled (g)	THF Insolubles (g/100ml)	THF Insolubles Rejection (%)	Event	Comments
Conc. Ran #5							
10/2/94 7:50	deash-46	0.9181	0.9259	0.0078	99.90	Start	Took initial permeate flux sample (deash-46) then began permeating to permeate collection barrel. Memebrane area for initial flux calculation = 0.1092 m2
10/2/94 7:55							
10/2/94 8:00							
10/2/94 8:05							
10/2/94 8:10							
10/2/94 8:15							
10/2/94 8:20							
10/2/94 8:25							
10/2/94 8:45							
10/2/94 8:50							
10/2/94 9:15							
10/2/94 9:25							
10/2/94 9:35							
10/2/94 9:40							
10/2/94 9:50							
10/2/94 9:55							
10/2/94 10:00							
10/2/94 10:05							
10/2/94 10:10							
10/2/94 10:25							
10/2/94 10:30							
10/2/94 10:40							
10/2/94 10:45							
10/2/94 10:50	deash-47	0.8773	0.8776	0.0003	100.00	End	Took final permeate flux sample (deash-47) then ceased concentration study. Module soaked overnight in room temp. toluene. Module inspection showed 12 plugged feed channels - membrane area for final flux calculation = 0.104 m2

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Attachment 7
Development of Analytical Tests for Coal Liquids and Solvent

Attachment 7 - Development of Analytical Tests for Coal Liquids and Solvent

CeraMem worked with Consol, Inc. on the analyses of initial feed, permeate and concentrate samples from each of the diafiltration/concentration runs. The goal was to develop test methods to analyze the samples in order to determine the quantities of starting coal liquid (O-13 reactor flash drum bottoms) and diluent (HRI's petroleum-based hydrotreated startup oil) in the process concentrates and permeates. The concentration of coal liquid in the concentrates and permeates is an important factor in analyzing the diafiltration/concentration process used in the project. The original approach, which was to distill the mixtures, thereby driving off the diluent first followed by the coal liquid, was not possible because the startup oil had a distillation curve almost identical to that of the particulate-free coal liquid. Therefore, as part of this program, alternative approaches were considered.

Consol and CeraMem, with assistance from Hydrocarbon Research, Inc., discussed several approaches to the analysis. First, the levels of asphaltenes and preasphaltenes in the coal liquid and diluent may be different enough to back calculate the amounts of coal liquid and diluent in each process stream. The measurements are straight forward, but the amounts of coal liquid and diluent calculated to be in each of the streams are dependent on the liquids being miscible. Other simple component measurements, such as C/H ratios, trace metals, nitrogen, and sulfur, were also reviewed; but the difference between the coal liquid and diluent was typically less than a factor of two and was deemed to be too small to use as an analytical marker. One other consideration was to use C^{13} isotope measurements as part of an involved analytical procedure. Although the procedure was not discussed much it was clear that it would be expensive and might not provide the desired results. Consequently, it was decided that Consol would start on the asphaltene and preasphaltene analyses in order to determine if the procedure would work.

The approach to developing the asphaltene/preasphaltene analytical method was to first determine the asphaltene and preasphaltene concentrations in the coal liquid and diluent and then determine if the feed mixture contained the calculated amount of asphaltenes and preasphaltenes based on the amounts in the coal liquid and diluent. If these tests proved that the coal liquid and diluent were significantly different and that the liquids were miscible so that the feed mixture asphaltene and preasphaltene concentrations could be calculated, then the approach might be applicable to calculating the concentration of coal liquid and diluent in the process permeates and concentrates from the diafiltration tests.

The particulate-free flash drum bottoms, startup oil, and initial feed mixture were analyzed in duplicate by Consol for asphaltene and preasphaltene concentrations. In general, the samples were first subjected to tetrahydrofuran (THF) solubility determination, followed by liquid column fractionation of the THF-soluble portions of each sample to determine the concentration of oil, asphaltene, and preasphaltene.

The results of these tests are reported in Table A7-1. The weight percent of THF-insolubles is slightly higher than that reported by HRI on the coal liquid (15%). The

weight percent of THF-insolubles in the mixture is lower than that reported by Imperial Oil (10.6%). It is unclear as to why this is so. Analyses of the THF-soluble fraction of each sample are reported as a percentage of the soluble fraction, not the whole sample. The concentrations of asphaltenes and preasphaltenes are both less than 5% for all three samples. Within the margin of error, there is no difference between the asphaltene or preasphaltene levels. It appears that due to the very low asphaltene and preasphaltene levels in this coal liquid, it would be very difficult to determine the actual concentrations of coal liquid and startup oil in the concentrates and permeates through the five diafiltration cycles.

Table A7-1.
Analysis of Coal Liquid Fractions

	% of Whole	Percentage of THF Soluble Fraction		
Sample ID	THF Insol.	Oils	Asphaltenes	Preasphaltenes
O-13 Bottoms	15.78 wt%	92.71 wt%	4.00 wt%	3.79 wt%
Start-up Oil	-	95.77 wt%	2.35 wt%	1.88 wt%
Initial Feed	8.72 wt%	92.89 wt%	4.15 wt%	3.00 wt%